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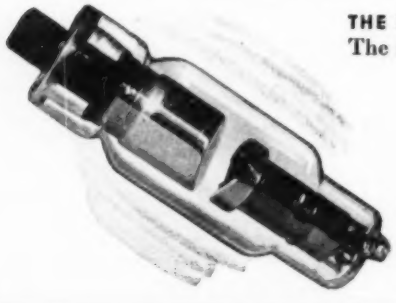

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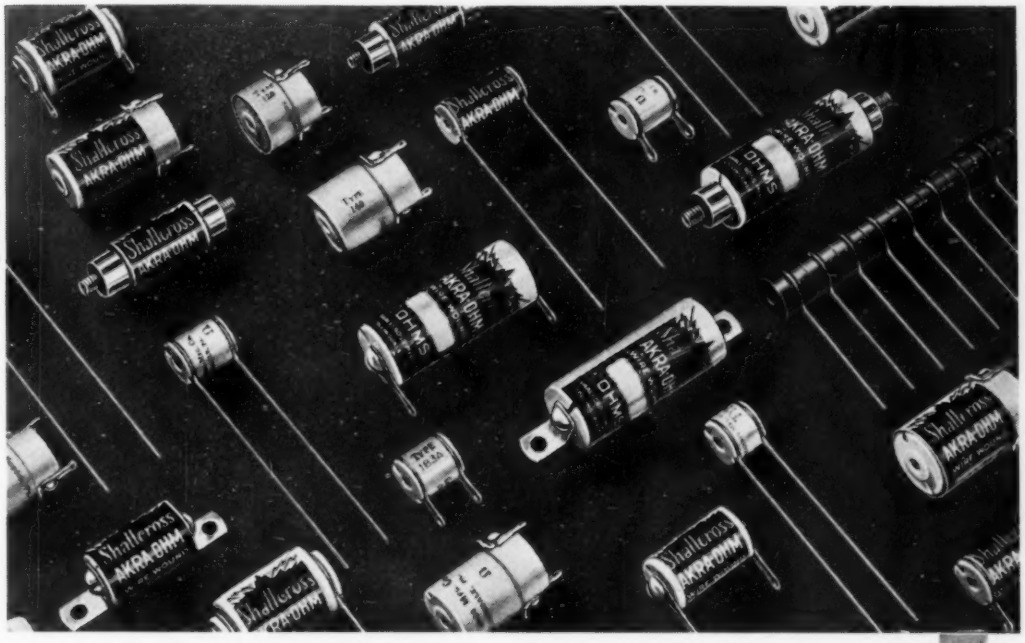

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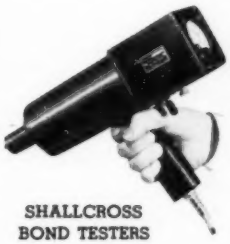

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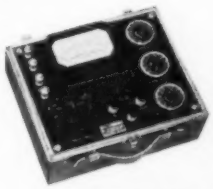

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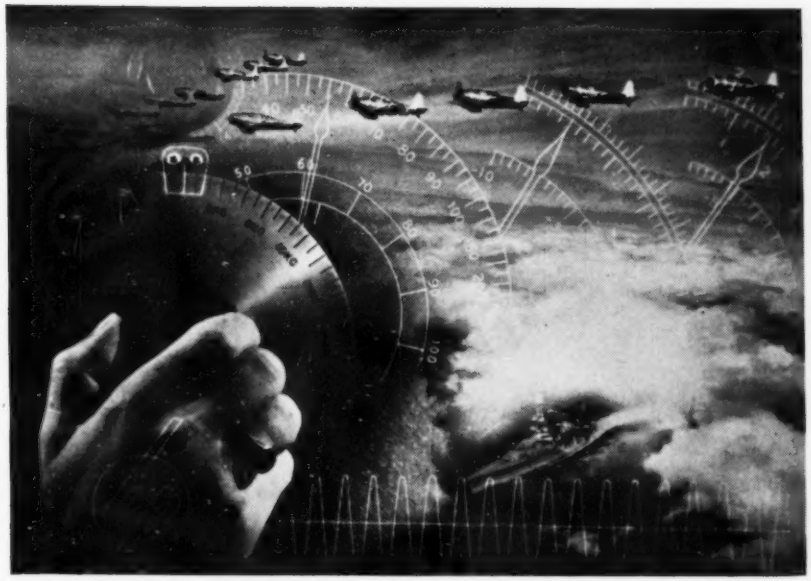

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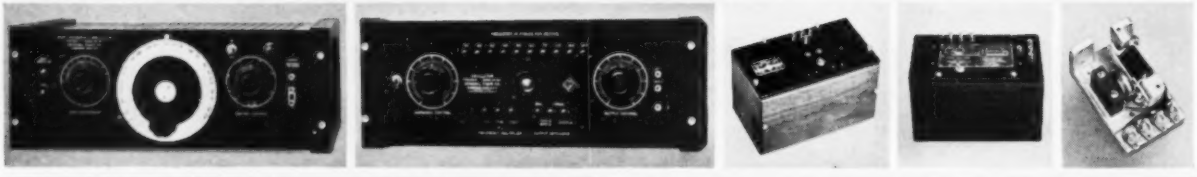

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Modern Spectrochemical Analysis

BY EDWIN K. JAYCOX

Bell Telephone Laboratories, Inc., Murray Hill, New Jersey

THE spectrograph, originally developed by the physicist, has become a most useful tool in the hands of the analytical chemist. Today few large analytical laboratories are without one. The instrument, with its attendant accessories, provides a rapid method for analyzing metals, alloys, minerals, ores, liquids, and gases, particularly for their metallic constituents and in some cases for their anions. Both emission and absorption spectra are important to the analyst. Important applications of the spectrograph to the analytical problems of research and industrial organizations will be discussed.

The spectrograph did not come into general use as an analytical tool until the early 1920's, although Kirchhoff and Bunsen saw the practicability of the method in 1860, when they published their paper entitled, "Chemical Analysis by Means of Spectral Observations." During the intervening years only a few enthusiasts like Lockyer, Roberts, Hartley, Leonard, Pollack, and de Gramont, kept the art alive. In spite of their persistent efforts to influence chemists to use spectrographic methods, they were quite generally ridiculed and the value of the method was recognized by only a few workers.

In 1922, Meggers, Kiess, and Stimson,[1] published their paper "Practical Spectrographic Analysis" and modern spectrochemical analysis was born. Under the stimulus of this paper and the backing of a high caliber scientific organization like the Bureau of Standards, the use of the spectrograph as an analytical tool increased rapidly. This is evidenced from the *Index to the Literature on Spectrochemical Analysis* by Meggers and Scribner.[2] In 1920, for example, only 5 papers were published concerning spectrochemical analysis, 4 of which were by de Gramont; whereas in 1930, 33 papers were published and in 1939, 170 papers, indicating an increasing interest in and use of spectrochemical analysis in industrial and research organizations.

APPARATUS

Spectrographs

There are today a number of manufacturers who can supply prism and grating spectrographs suitable for all kinds of analytical work. Up to a few years ago, only prism instruments were generally available. The difficulties of ruling gratings were so great and the process so slow that grating spectrographs were custom built and therefore expensive. With recent improvements in ruling methods glass and metal gratings can now be rapidly and accurately made, so that reasonably priced instruments are now on the market. Although there has been much controversy over the merits of prisms *versus* those of gratings for analytical work, the writer is convinced that either type will do an equally good

job in the field of spectrochemical analysis for both emission and absorption when properly handled. For discussions of this controversy the reader is referred to Harrison[3] and Slavin.[4]

Sources

Several types of sources are capable of exciting emission spectra. These exciting agents are flames, arcs, sparks, and gaseous discharges.

The direct current arc is probably the most widely used of all sources. It is capable of exciting the spectrum of all metals and in addition arsenic, boron, carbon, phosphorous, and silicon. It is the most sensitive source for most of the metals and for this reason it is particularly adapted for qualitative analyses. It is used a great deal for quantitative work but is not as reproducible as the spark or alternating current arc.

Another type of arc is the high voltage alternating current arc proposed by Duffendack and Wolfe.[5] Our own modification of their circuit[6] has proved to be a very satisfactory source, especially for the analysis of liquid samples dried on the ends of flat top graphite electrodes. It is steadier and more reproducible than the direct current arc and in some cases more sensitive. It combines some of the precision of the condensed spark with the sensitivity of the direct current arc. It has also been used successfully in our laboratory for the analysis of ceramic materials.

Spark sources are usually more reproducible and precise than arcs. They lack the sensitivity of the arc for most elements but are capable of exciting some elements, notably the gases and certain anions not excited by the arc. In the spark higher temperatures are attained in the vapor column between the electrodes, which give rise to the emission of light from higher energy states of the atom. The electrodes themselves remain relatively cool. For this reason, it is practical to spark between self-electrodes of the metal or alloy, even for low melting point metals and alloys. Finished products can be sparked against a counter electrode with only a slight etching action on the surface and without appreciable damage to the product. One disadvantage of the spark in spectrochemical analysis is the small amount of sample consumed, which in many cases is not representative of the sample if there is segregation of the elements present.

Most spectrochemical laboratories today are equipped with spark circuits having power outputs of 0.5 to 10 kva and peak voltages of

FIG. 1. General view of spectrochemical laboratory.

10,000 to 40,000 volts. Means for varying the capacity and inductance of the circuit are generally provided to control the character of the spark. Additional control is sometimes employed which utilizes only certain portions of the discharge wave, thereby attaining a more reproducible source. This control, devised by Fuessner[7] and further improved by Kaiser,[8] is obtained by placing a synchronous rotating gap in series with the spark gap. This allows the condenser to be discharged only at the moment when it is at maximum charge in each half-cycle and assures that a predetermined type of discharge can be secured at any time.

Hassler and Dietert[9] have recently described an all-purpose circuit and apparatus, the characteristics of which can be varied from that of an arc through numerous intermediate stages, including that of the "controlled" spark and that of a highly condensed spark. A circuit such as this should be a great asset in a spectrochemical laboratory, as it enables the analyst to have available, in a single unit, the characteristic spark or arc needed for the problem on hand.

ELECTRODES

For many analyses, especially with spark sources, self-electrodes of the sample are used. The shape and size of these electrodes are carefully controlled, particularly in quantitative analyses. Sometimes pointed counter electrodes of the same base metal or of graphite are used to spark against a flat surface of the sample.

For most analyses with the arc, use is made of graphite electrodes, the lower one in the form of a cup to carry the sample and the upper one a blunt or pointed counter. The graphite cups may be varied in shape and size to accommodate different kinds of samples, i.e., millings, filings, powders, or liquids. Spectrographic graphite rods are available in several sizes from $\frac{1}{16}''$ in diameter to $\frac{5}{16}''$ in diameter, and in two grades, ordinary and purified. The ordinary grade usually contains appreciable amounts of silver, boron, calcium, copper, potassium, iron, sodium, magnesium, lead, silicon, titanium, and sometimes other elements. It is therefore not suited for qualitative or quantitative work involving trace amounts of these elements, unless it is first purified. Pre-arcing the electrodes at high currents will get rid of some of the more volatile elements. Purification by the method of Staud and Ruehle,[10] or by the method of Owens,[11] yields electrodes of satisfactory purity. Very pure graphite rods may be obtained from the National Carbon Company or the Dow Chemical Company. The use of graphite electrodes does have one drawback, the production of the cyanogen bands which tends to mask some important lines in the region from 3500A to 4200A. In order to avoid the cyanogen bands, electrodes of copper, silver, or other metals are sometimes used.

PHOTOMETRY

Precise quantitative spectrochemical analyses require some form of densitometer to measure the densities of spectral lines. The fundamental features of a densitometer are a light source which provides a measuring beam passing through a restricted area on the plate or film, and a means of comparing the amount of light traversing this area with that of a similar area on a clear portion of the plate or some standard medium. There are available subjective visual types of densitometers and objective types utilizing photoelectric cells or thermopiles which actuate a galvanometer or other current measuring device. Of these the objective type is the best for the needs of the spectrochemical analyst because it is inherently more accurate and free from subjective errors. The instruments may be either recording or spotting, but the nature of the measurements involved is such that the spotting type will be found more convenient and faster. The optical and mechanical details of available instruments differ in many respects but the fundamental procedure followed in measuring relative densities is essentially the same.

$$\text{Line density, } d_l = \log (I_0/I),$$

where I_0 is the intensity of the incident beam and I is the intensity of the emergent beam. Translated into terms of galvanometer readings

$$d_l = \log \frac{I_0}{I} = \log \frac{G - G_0}{G_l - G_0},$$

where G, G_l, and G_0 are, respectively, the gal-

vanometer readings corresponding to I_0, I, and zero (total blackness). If the galvanometer scale is adjusted so that G_0 is always zero and G is always at a definite setting, then

$$d_l = \log \frac{K}{G_l} = -k \log G_l.$$

Since d is a function of $\log G_l$ and k is constant for a given set of determinations, for the purpose of comparison, relative density can be represented simply by $\log G_l$.

If the relation between density and log intensity were linear it would be possible to correlate density and concentration directly. Also it would be possible to correct for background d_b by subtracting d_b from d_l. The d-*vs.*-$\log I$ relationship, however, is linear only for a limited range of densities; hence it is necessary to determine the shape of this curve for the type of plate used. Theoretically this should be done for each plate, but in practice it is frequently permissible to determine the density range over which the relationship is linear for the type of plate and processing used, and restrict the densities used to this range.

The calibration of plates may be accomplished by impressing a known intensity pattern on the plate. This may be done conveniently with a stepped sector, a stepped wedge, a stepped slit, or other means. From this intensity pattern the curve of d-*vs.*-$\log_a I$ is obtained, or more commonly G_l-*vs.*-$\log_a RE$, where a is the step ratio and RE is the relative exposure, since in most cases it is unnecessary to obtain actual intensities.

SPECTROCHEMICAL LABORATORY AT MURRAY HILL

Figures 1, 2, and 3 are pictures of our spectrochemical laboratory at Murray Hill, New Jersey. Figure 1 is a general view showing the arrangement of the principal pieces of apparatus. Figure 2 shows in more detail the layout of our medium Zeiss spectrograph, the arc and spark stand, the fume hood, the external optics, a Hilger Spekker photometer, arc and spark power sources and controls, a small Bausch and Lomb Littrow spectrograph, and the sample preparation desk. Figure 3 shows our Zeiss densitometer, a Judd Lewis comparator, and part of the table used for the interpretation of plates.

Other equipment not shown in the photographs includes a fully equipped dark room,

FIG. 2. Zeiss spectrograph and accessory apparatus.

chemical hood, adequate services such as gas, high and low pressure air, vacuum line, oxygen, distilled water, and electric power. The laboratory was designed for maximum efficiency and convenience and during the past year has been found to have lived up to the expectations of the designers.

APPLICATIONS

Qualitative Analysis

From the standpoint of the techniques involved, qualitative spectrochemical analyses are simpler than quantitatives, but the interpretation of the resulting spectrograms may often be far more difficult. This is especially true if the analyst is to give more information than simply whether elements are present or absent. A qualitative analysis to be of utmost value should classify those elements found according to the order of magnitude in which they are present. This of course requires experience and the exercise of considerable judgment. At the Bell Telephone Laboratories we have found it advantageous to classify the elements detected as follows:

Principal components	>10 percent
Major component	>1 percent
Minor component	0.10–3 percent
Impurities	0.01–0.3 percent
Traces	<0.03 percent
Faint traces	<0.003 percent
Not detected	Lowest limit detectable.

It should be emphasized that such estimates are only intended to act as a rough guide and should never be considered as quantitative determinations. Samples may be received in all sorts of forms and quantities and may contain a variety of elements, some of which influence the excitation of the lines of other elements. Analyses involving large amounts of the alkali metals, or such metals as tungsten, thorium, uranium, and molybdenum, often affect the excitation of other metals to a very marked degree. The amount of sample consumed varies from less than a microgram to several milligrams in different analyses, so that in one case a line of given density might indicate a major component, and in another only a trace. All of the above classifications are seldom used in a single analysis. Classifications and

FIG. 3. Densitometer and plate interpretation table.

their limits are chosen to give the best interpretation of the results and to convey to the engineer submitting the sample the maximum information desired.

The direct current arc is the source usually used for most qualitative analyses in our laboratories. The sample in whatever form, solid, powder, or solution, is introduced into the positive crater of the arc and arced at 4 to 10 amperes for 60 seconds, or until completely consumed, the exact procedure followed depending upon the character of the sample. It is a good practice, in addition, to obtain a "moving plate" [12] spectrogram on another portion of the sample. This helps in the detection of the various components and sometimes shows up elements that might be missed in a single exposure. A reference spectrogram of iron or copper is usually taken alongside of the spectrogram of the sample by means of a Hartmann diaphragm to facilitate the identification of lines. If some particular element is sought, a spectrogram of this element may also be taken in juxtaposition to that of the sample.

The identification of all of the elements in a sample requires that some systematic procedure be followed, otherwise important components might easily be overlooked. The procedure followed in our laboratories is to look specifically for each of thirty-six elements which are always sought, and for certain additional elements depending upon the nature of the major components involved. At the Bell Telephone Laboratories, as an aid to the identification of the elements, we have built up a library of reference

FIG. 4. Reference plate—large Hilger spectrograph.

plates for each of our spectrographs. These reference plates have photographed on them the spectrum of the reference element in juxtaposition to an iron or copper spectrum.

Figure 4 shows a portion of a typical multiple element reference plate for our large Hilger instrument, showing the principal lines of Cd, Co, Cr, Cu, Mg, Mn, and Mo, together with the iron arc spectrum. In this case the spectra of the reference elements were taken through a step sector with the iron spectrum superposed near the bottom where the lines are weakest. This was done so that some of the weaker lines could be registered and at the same time the stronger lines would not be overexposed in the neighborhood of the iron spectrum where comparisons with experimental plates would be made. Figure 5 is a typical reference plate for Ba, Be, and Bi, for our medium Zeiss instrument with copper in juxtaposition with each element.

In making an analysis plate, iron or copper is registered in juxtaposition above the spectrum of the sample. This plate can then be laid on the reference plate over a suitable viewing box, with the iron or copper spectra on reference and test plates coinciding. With the use of a 6X magnifier it is a simple matter to determine whether an element is present in the sample. Instead of a simple viewing box a comparator such as the Judd Lewis may be used. The principal line or "raie ultime" is first sought and if found, other lines of the element are sought for confirmation. The spectroscopist at this point must be on his guard for lines of other elements which are close to the lines sought, and the spectrogram should be checked for such interfering elements.

When the elements present in the sample have been identified, the task of estimating the amounts begins. There is no better method available than that the analyst acquire experience in evaluating spectrograms so that this judgment can be backed by this experience. Skill can be

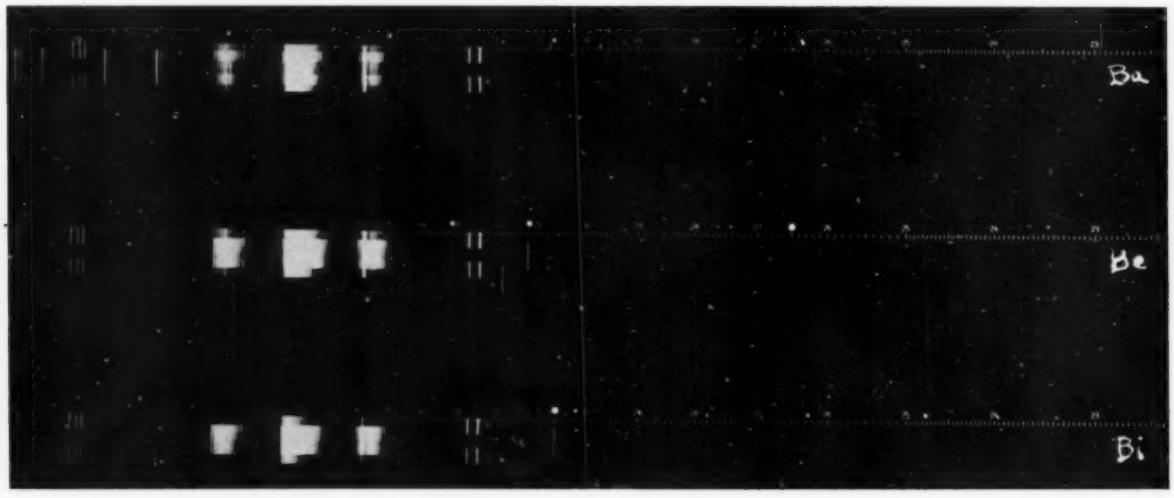

FIG. 5. Reference plate—medium Zeiss spectrograph.

acquired by the beginner by running samples and comparing his estimates with those of an experienced spectroscopist or by running quantitatively analyzed samples as unknowns, and comparing his estimates with the analysis.

In a research organization like the Bell Telephone Laboratories, the spectrochemical laboratories are called upon to make qualitative analyses on all types of materials including metals and alloys, ceramics, precipitates, reagents, organic materials, ashes, minute deposits, surface contaminants, and gases. No one particular technique is suited for analyzing all such materials, and new techniques and modifications of old techniques must constantly be devised to fit particular applications both in sampling and exciting.

When a complete qualitative analysis is to be made, metals and alloys are usually introduced into the arc as millings or drillings, or in some cases where the sample is very small the entire sample may be used. Samples are usually arced in the d.c. arc at current values depending upon the nature of the sample, low melting point materials at the lower currents, and high melting point materials at higher current values. Metals such as tungsten, molybdenum, thorium, and uranium and their compounds are exceptions. These are run at very low currents, 2 to 4 amperes, to avoid blackening the plate with the continuous background spectra characteristic of these elements. When two samples are to be compared for differences in composition, more reproducible spectra can be obtained if the samples are taken into solution in a suitable reagent and run as solutions, or as dried salts mixed with pure carbon dust.

Figure 6 illustrates an interesting analysis where identification of a particular alloy and differences in composition were involved. One of the operating telephone companies had been troubled by having lengths of lead-antimony covered cable stolen from reels left on job locations. A suspect was found who had in his possession large quantities of melted up lead. The suspect claimed he had obtained the lead by purchase of old cable from one of the power companies but could not substantiate his statement. The police secured samples of the melted lead from the suspect and also samples of cable used by nearby power companies. These samples were sent to us for analysis. Referring to Fig. 6, spectrogram No. 1 is a piece of standard lead-antimony telephone cable sheath. Samples No. 2 and No. 3 are from power company cable sheaths. Sample No. 4 is some of the melted lead seized from the suspect, and No. 5 is lead scraped from the floor of the room where the melting had taken place. A glance at the spectrograms shows that samples 1, 4, and 5 are the same alloy, and that they are different from samples No. 2 and No. 3. The Bell System cable is identified by its high antimony content, the power company's cable by its high bismuth content. In addition, silver and copper are higher in the telephone cable. From this evidence there is no question but that the lead found on the suspect's premises was from telephone cable.

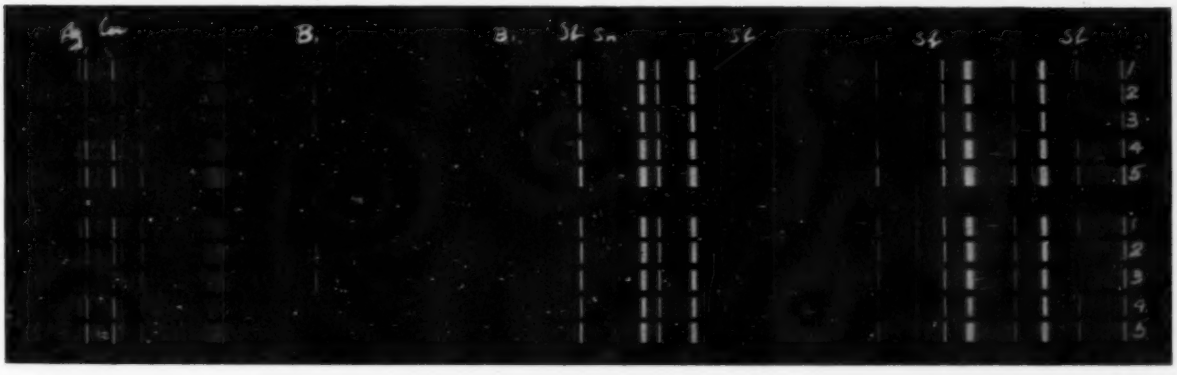

FIG. 6. Lead cable sheaths.

Figure 7 is a plate taken for a complete qualitative analysis and comparison of four samples of organic ash. The ashes were mixed with equal parts of pure carbon dust, and aliquots were placed in the craters of graphite cup electrodes and arced. The samples were all similar. Major components were Mg and Na, minor components were Al, Ca, Fe, and Si, with some impurities and traces. The only differences observed were in Ba and Sn in the classifications of impurities, traces, or not detected.

The plates shown illustrate only a few examples of qualitative analyses. Among other materials frequently analyzed at the Bell Telephone Laboratories are precipitates obtained in chemical reactions, which aid the chemist in determining whether his reactions are what he hopes them to be or whether some extraneous element is present which he has not taken into account. Organic materials and ashes are frequently analyzed for metallic components. Surface contaminations and deposits from many sources require analysis. A method for the identification of relay contact metals has been employed here for several years. The contact is rubbed with a piece of filter paper impregnated with 400-mesh Al_2O_3. The paper is rolled into a small ball placed in a graphite cup and arced. The principal lines of all the noble metals used in relay contacts can be identified in this manner without injuring the contact or even removing the relay from its rack, and with only a momentary interruption of service.

Another type of qualitative analysis is the identification of gases used in various applications. These may be contained in suitable vessels for excitation or can be sealed in Geissler tubes for examination. On this type of work our system of reference plates partially breaks down, because the spectrum obtained from many of the gases depends upon the pressure, the type of excitation, and the geometry of its envelope, so in most cases it becomes necessary to determine the wavelengths of most of the recorded lines in order to make a complete analysis.

The examples cited give some idea of the usefulness of qualitative spectrochemical analyses. There are of course many other specific applications for which the spectrograph can be adapted.

QUANTITATIVE ANALYSIS

The total sample consumed in a spectrochemical analysis is usually very small. For this reason all specimens for analysis should be considered as non-homogeneous in composition unless there is positive evidence to the contrary, such as shown by physical tests, by the method of production, or by repeated analyses on different aliquots. If such evidence cannot be obtained it is necessary to mill the sample and either mix the millings thoroughly or take them into a solution. Bricquetting of finely milled samples is another method of obtaining a uniform sample.

It is very important that standards and samples be in exactly the same physical and chemical condition prior to excitation. If self-electrodes of metals or alloys are used, with either the spark or arc, it is essential that the size, shape and surface condition be exactly reproduced, otherwise the excitation of the spectra will not be comparable. The over-all composition of the standards and sample must not be too different since the presence of several percent of an element in a sample frequently affects the excitation of other elements. For example, if one wants to determine Mg, of the order of 0.2 percent, in an aluminum alloy containing approximately 8 percent Cu and 1.5 percent Si, the standards should contain these elements in about the same order of magnitude. Although the non-metals themselves are not excited they often have a pronounced effect upon the excitation of the metals, therefore the state of combination of the elements should be reproduced as closely as possible.

The excitation conditions, whether flame, arc, spark, or Geissler discharge, must be as closely controlled as possible. Besides holding the circuit conditions constant, control is often maintained by secondary agents. For example, with ceramic materials, carbon dust or some other buffer material is mixed with both standards and samples to cause more even sparking or arcing. Iron or copper electrodes are sometimes oxidized and only a small area filed clean to prevent the arc or spark from climbing away from the desired point.

Quantitative spectrochemical analyses fall

FIG. 7. Analysis of organic ashes.

into two general classes, namely routine and non-routine analyses. Routine analyses are used mainly in industrial laboratories for the control of production of metals, alloys, and other products, or for checks on the composition of raw materials purchased from outside sources. Methods used for this type of work have usually been developed to meet a particular need and are rapid and accurate.

The most common of routine analyses is the analysis of metals and alloys. Spark excitation is usually used, with self-electrodes. Frequently electrodes of proper size and shape are cast directly from the melt for which they are the control. Routine methods, whether with self-electrodes, solutions, or powders, are thoroughly standardized, sometimes after weeks of research. Size and form of sample, excitation conditions, the manner of recording spectra, the choice of spectral lines used, and the interpretation of the spectra are all carefully studied and the precision of the method determined. Routine analysts then take over, adhering closely to the prescribed techniques. Such methods are usually rapid and accurate, yielding precisions in some cases as good as ± 1–2 percent.

Non-routine methods are characterized by slower speed and moderate precision. Since they are frequently of non-recurring nature, there is not the justification for spending a great deal of time in developing a method for a specific analysis which might never be used again. Consequently, general methods are developed, which have a wide application and which can be readily modified to meet a particular need. Such methods are usually based on the use of solutions and powders.

In a large research organization like ours, interested in a wide variety of materials, the spectrochemical laboratories must be prepared to handle almost any conceivable type of analysis. We are equipped to do routine analyses on the more commonly used materials and for some of these we employ spark excitation with self-electrodes and carefully analyzed standards. The greater part of our work, however, is of non-routine nature. In order to handle, readily, the diverse types of analyses demanded of us, we keep on hand stocks of pure metals, standard solutions, salts, oxides, etc., from which we can quickly make solution or powdered standards.

Standards employing solutions or powders as a base can be made up very accurately. Solutions have a marked advantage over other methods for obtaining homogeneous samples. Relatively large amounts of the specimen can be taken into solution reducing the danger of segregation of elements. At times it is impractical to put the sample into solution, especially with such materials as ceramics. These are usually pulverized to a fine powder and mixed with pure carbon dust or other buffer materials in order to obtain more uniform excitation in the arc or spark.[13,14]

To make a quantitative analysis, spectrograms of the samples and standards are recorded on the same plate. Excitation conditions are kept constant and the amount of light entering the spectrograph is controlled by means of a radial sector, neutral filter, or diaphragm, so that the densities of the lines to be used will be in the proper density range for densitometry.

The interpretation of the plate may be carried out by either of two procedures, the comparison standard or internal standard methods. In the comparison standard method the density of the line of element x sought in the sample is compared with the densities of x in the standards. If the plate has been calibrated, d_x is obtained in terms of $\log_a RE_x$ and a curve of percent con-

centration of x-*vs.*-$\log_a RE_x$ is plotted for the standards. Values of x for the sample are obtained from this plot. In cases where the plate has not been calibrated, the relationship of concentration of x-*vs.*-$\log G_x$ is obtained from the standards and the concentration of x in the sample obtained from a plot of this relationship.

The internal standard method is an extension of the comparison standard method, wherein a suitable line of the major component or that of an added element is used for control. If it is assumed that the density of all lines in a spectrogram varies in the same ratio as the exposure becomes greater or less, then some line of an element which is present to an equal amount in all samples can be used as a control. This is the theory of Gerlach[15] and is true within certain limits.

If y is the internal control element, a plot is made of concentration of x-*vs.*-$\log_a RE_x - \log_a RE_z$, (or $\log G_x - \log G_y$ if plate calibration is not used) and the amount of x in the sample obtained from the curve.

Figure 8 is a typical plate for the analysis of a zinc base alloy for Cd, Fe, Mg, Sn, and Pb. Standards and samples in this case were in the form of chloride solutions with the concentration of zinc plus aluminum = 100 mg/ml. 0.1 ml aliquots of samples and standards were pipetted to waterproofed graphite cups, dried in an aluminum heating block at 110°C, and arced in the d.c. arc at 8 amperes for 1 minute. Spectrograms of 4 aliquots of the sample, and a graduated series of standards, containing from 0.001 to 0.10 percent of each element sought, are recorded on the plate. This analysis was run using the comparison standard method, as zinc, because of its high volatility, is unsuited for internal control. The average precision of this method is ±90 parts/1000. Equally good results can be obtained by the use of the dry nitrate salt with carbon dust as a buffer and a.c. arc excitation.

A typical rapid control method is the analysis of lead alloys for calcium. Solid standards and samples are used with controlled spark excitation between a flat surface of the alloy and a pointed graphite counter electrode. A lead line is used for internal control in the interpretation of the spectra. Suitable line pairs are:

Ca 3933.7/Pb 3739.9,
Ca 3968.5/Pb 3671.4,
Ca 4226.7/Pb 4019.6.

This is a rapid and accurate method, suitable for routine control analysis. In a test run, three samples were analyzed (3 checks on each) on the following schedule: Excitation 9 minutes, plate processing 5 minutes; plate drying 9 minutes, densitometry 9 minutes; calculations 8 minutes;

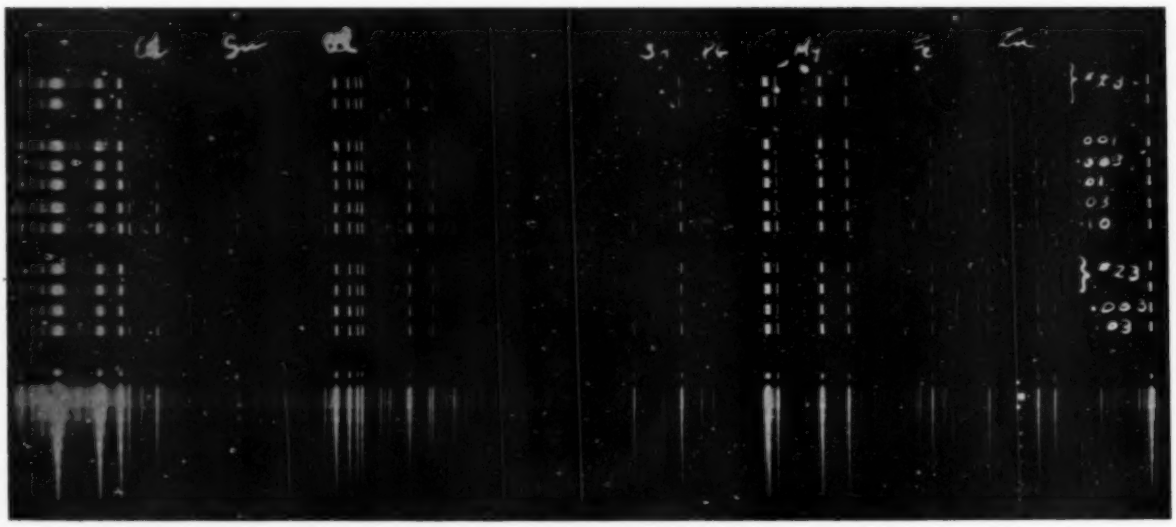

FIG. 8. Analysis of zinc base alloy.

FIG. 9. Analysis of aluminum base alloy.

total 40 minutes. Increased speed will result if rapid processing apparatus such as used by Vincent and Sawyer[16] is employed and if mechanical aids in the calculations are installed. The results in Table I indicate the accuracy of the method.

TABLE I.

Sample	Ca found Spectrochemical	Ca found Chemical	Dev.
1*T*	0.032	0.034	−0.002
1*W*	0.037	0.037	0.000
1*Y*	0.042	0.041	+0.001
2*A*	0.030	0.030	0.000
2*C*	0.034	0.034	0.000
2*H*	0.030	0.033	−0.003
2*J*	0.024	0.024	0.000
2*M*	0.031	0.031	0.000
		Average	±0.0008

The accuracy obtained in this analysis is exceptionally good. It should be pointed out, however, that such results can be obtained only after all the factors entering into the procedure have been thoroughly studied and the exact techniques selected. The general procedure is applicable to the analysis of other elements in lead alloys as well as to the analysis of impurities in other base alloys, whenever a reliable set of solid standards is available.

Figure 9 is a plate used in the analysis of an aluminum base alloy for Cu, Fe, Mg, Mn, Ti, and Pb. In this case the spectra were taken through a three-step diaphragm placed in front of the spectrograph slit, which transmits, respectively, 100 percent, 20 percent, and 4 percent of the light. Such a device impresses upon the plate three sets of spectra for a single exposure and permits a wider choice of lines that will be in a proper density range for densitometry than will a single spectrogram. This is especially helpful in cases where six to eight elements in a sample are sought. It also permits the calibration of the plate since the ratio of the exposures remains fixed. Because of the large increment between steps it is not as accurate as a step sector with six to eight steps, but permits a check on whether the spectral response curve is a straight line in any particular region.

ABSORPTION

Another useful application of the spectrograph in an analytical laboratory is for the identification and determination of the amounts of organic and inorganic compounds present in

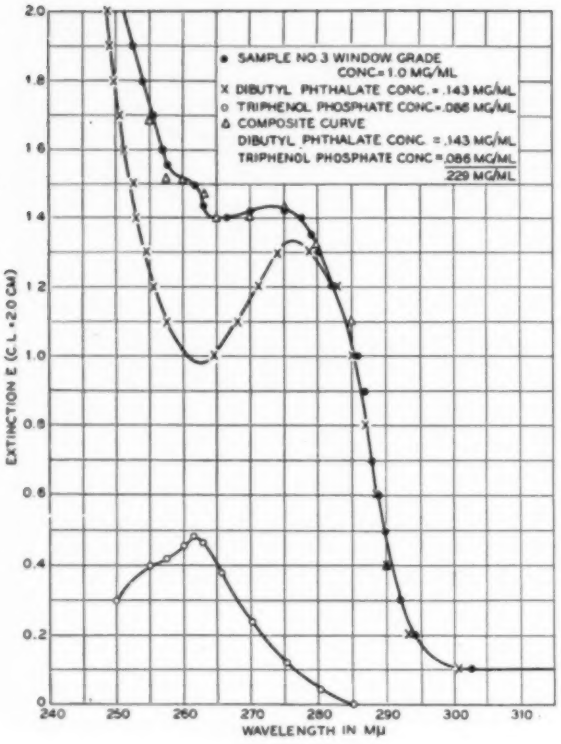

FIG. 10. Analysis of cellulose acetate.

many types of samples, by the measurement of their absorption characteristics. As a qualitative tool the identification of compounds is much less rapid than the identification of elements by emission spectra. Even though the compound may have a characteristic absorption band it is not always easy to identify without a thorough search through the literature. As a quantitative tool, absorption is much more valuable, for in this case the identity of the compound is usually known and quantitative methods can be applied to determine the amount present.

At the Bell Telephone Laboratories we use a Hilger Spekker photometer in conjunction with a Zeiss medium spectrograph to measure the absorption characteristics of various compounds.

We were confronted with the problem of determining the amounts of certain plasticizers in cellulose acetate plastics. Chemical methods were available for identifying most of the commonly used plasticizers, but in most cases there were no means of making quantitative determinations. A number of these organic compounds have absorption bands in the range of 240 mμ to 600 mμ. For such compounds spectrophotometric measurements can yield important information both for the identification and the determination of the amounts present.

Figure 10 illustrates how an analysis was made on one sample of cellulose acetate. The total plasticizer content had been found by chemical methods to be 23.92 percent of the total sample weight. The plasticizers identified were dibutyl phthalate and triphenol phosphate, both of which have characteristic absorption curves and obey Beer's law; that is, the amount of absorption is proportional to the concentration. The curve through the dots shows the absorption characteristic of the sample at a concentration of 1.0 mg/ml in a solvent made up of 9 parts of chloroform and 1 part of absolute alcohol. The crosses show the absorption curve for dibutyl phthalate at a concentration of 0.143 mg/ml, and the circles triphenol phosphate at 0.086 mg/ml.

An examination of the absorption curves for the sample, dibutyl phthalate and triphenol phosphate, shows that at wave-lengths above 285 mμ, triphenol phosphate contributes nothing to the absorption of the sample and hence any absorption beyond this point is due entirely to the dibutyl phthalate. The concentration of dibutyl phthalate can be obtained from a plot of extinction E *versus* concentration at 288 mμ, which in this case was 0.143 mg/ml. The difference in extinction E between the absorption curves for the sample and dibutyl phthalate, at say 260 mμ, is the amount of absorption caused by the triphenol phosphate. The concentration of triphenol phosphate can be obtained from a plot of E *versus* the concentration of triphenol phosphate at 260 mμ, which in this case is 0.086 mg/ml. The sum of the concentrations of the two components is 0.143 mg/ml+0.086 mg/ml or 0.229 mg/ml, or 22.9 percent of total weight of sample. This compares with 23.9 percent obtained by chemical means, a reasonably good check. The curve through the triangles in Fig. 10 is the composite of dibutyl phthalate and triphenol phosphate at concentrations of 0.143 mg/ml and 0.086 mg/ml, respectively, and fits the experimental curve for the sample quite well.

The above example is only one of many applications of absorption apparatus in an analytical

laboratory. Spectrophotometric absorption data is a valuable tool, both in its own right, and as companion tool to other methods, for the identification and determination of amounts present of many organic compounds in various types of samples.

CONCLUSIONS AND ACKNOWLEDGMENTS

The spectrograph with its attendant auxiliary apparatus has become an indispensable tool in most industrial and research chemical laboratories. Its applications to chemical analysis are wide and varied, in both the fields of emission and absorption spectra, not to mention its usefulness in the field of pure physical phenomena. No other single instrument has such widespread scope.

The author wishes to acknowledge his indebtedness to Mr. Werner Hartmann and Miss B. E. Prescott for their aid in obtaining some of the data used in this paper; and to Mr. A. E. Ruehle, who played an important part in developing some of our fundamental methods and organizing the laboratories described.

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(16) H. B. Vincent and R. A. Sawyer, Spectrochimica Acta **1**, 131 (1939).

Calendar of Meetings

December

27 American Physical Society, Pasadena, California

January

14–15 American Physical Society, New York, New York
14–15 American Association of Physics Teachers, New York, New York
14–15 Electron Microscope Society of America, New York, New York
25–27 Institute of Aeronautical Sciences, New York, New York
31–February 2 American Society of Heating and Ventilating Engineers, New York, New York

February

14–17 Technical Association of the Pulp and Paper Industry, New York, New York
18–19 American Philosophical Society, Philadelphia, Pennsylvania
25–26 American Physical Society, Metropolitan Section, Brooklyn, New York
26 American Mathematical Society, New York, New York

March

2– 4 Optical Society of America, New York, New York

April

12–15 Electrochemical Society, Milwaukee, Wisconsin
20–22 American Philosophical Society, Philadelphia, Pennsylvania
27–29 American Physical Society, Pittsburgh, Pennsylvania
28–29 American Mathematical Society, New York, New York
28–29 American Mathematical Society, Chicago, Illinois
29 American Mathematical Society, Berkeley, California

May

12–13 Acoustical Society of America, New York, New York

Résumés of Recent Research

Conduction of Electricity in Highly Insulating Liquids

Investigations have recently been made by Chia-Shan Pao[1] to obtain more precise information as to the exact character of the conductivity found in insulating liquids. The liquids studied were liquid oxygen and isooctane. In these experiments it was necessary to reduce, by drastic purifying methods, the already small conductivity in isooctane. This was done principally by refluxing it for days in contact with metallic sodium and then transferring it in a closed system to the cell in which the conductivity was measured. In the case of liquid oxygen, commercial oxygen was condensed in a carefully cleaned vessel; no further purification was attempted. The lowest conductivity thus obtained is 3×10^{-20} mho/cm for isooctane and 0.2×10^{-20} mho/cm for liquid oxygen.

The possibility of a thermionic origin for the observed conductivity in these liquids seems ruled out by the present results. For a given field, the conductivity is not a constant, as it should be if it were of thermionic origin at the cathode, but increases with volume. In addition the conductivity is independent of the direction of the field; this is unaccountable on the thermionic theory. That the observed conductivity is caused by cosmic rays also seems improbable. Shielding the measuring cell with three to four inches of lead does not affect the conductivity; this is in agreement with the findings of Jaffé, Rogozinski, and Bialobrewski. Again, when the current I through the liquid is measured as a function of the field X in the presence of gamma-rays, the relation between I and X is so different that it is difficult to accept the cosmic-ray hypothesis for the natural conductivity. The relation between log I and X^1 is linear, a result which is predicted by the field dissociation theory. However, the slope of the line decreases 13 percent when the temperature changes from 309.5°K to 195°K, whereas a direct interpretation of the theory would imply a 38.2 percent increase, a result not anticipated by the simple theory.

The gamma-rays from radium produce a conductivity in isooctane and liquid oxygen whose current-field characteristics are in satisfactory agreement with Jaffé's theory of columnar ionization. The reciprocal of the current becomes a linear function of the reciprocal of the field for fields exceeding 10,000 volts/cm in the case of isooctane and for fields exceeding 6000 volts/cm in the case of liquid oxygen. The same curve also indicates that at fields of the order 10,000 volts/cm the current in liquid oxygen is further from its saturation value than in the case of isooctane. For a given field the relation between the current and the intensity of radiation is linear, a fact which shows that the liquid chamber can replace the ordinary gas chamber to measure different amounts of radiations.

The Structure of Liquid Xenon

The structure of liquid xenon has been investigated using the x-ray diffraction technique.[1] Ag $K\alpha$ radiation, monochromatized by reflection from a rocksalt crystal, was incident on a thin-walled glass capillary containing the liquid xenon. The diffraction pattern was recorded on a film mounted on a radius of 5.73 cm with the sample as center.

Patterns were obtained under three sets of conditions:

Run	Pressure in atmos.	Temp. °C	Sp. Vol. cc/g
1	1	−110	0.324
2	2.5	− 90	0.339
3	130	− 90	0.324

where it is seen that runs 1 and 3 are at the same specific volume and runs 2 and 3 are at the same temperature.

The structure of the liquid determined by a Fourier analysis of these patterns is shown in Fig. 1 where the relative probability W of finding an atom at some distance from a central atom is plotted against the distance r in A. This relative probability is based on an average probability of one if the liquid were a continuum and shows, therefore, the percentage deviation of the actual

[1] Chia-Shan Pao, Phys. Rev. **64**, 60 (1943)

[1] J. A. Campbell and J. H. Hildebrand, J. Chem. Phys. **11**, 334 (1943).

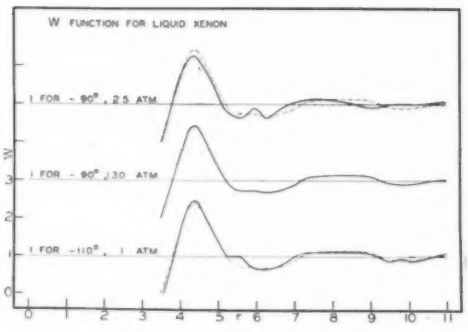


FIG. 1.

liquid from a continuum, with the peaks indicating a high probability and the valleys a lower one. The dotted lines are superimpositions of the curve for run 3 on those for runs 1 and 2. It is obvious that the structures found under the conditions of runs 1 and 3 are much more similar than those found in 2 and 3. That is, specific volume is much more important in determining the structure of liquid xenon than is temperature.

The number of nearest neighbors and their average distance from the central atom is listed below together with the distance of closest approach of two atoms, r_{min}.

Run	r_{min}	Nearest neighbors No.	Pos.
1	3.60	8.5	4.43
2	3.50	8.3	4.50
3	3.45	9.0	4.47

A comparison of the structure of liquid argon[2] and liquid xenon shows the two to be very similar when the reduced temperature is 0.604 for argon and 0.564 for xenon and the reduced volume is 0.384 for argon and 0.375 for xenon. It is thought that the reduced values for argon must be greater than those for xenon in order to overcome the effect of the greater kinetic energy of the xenon due to its higher absolute temperature.

[2] A. Eisenstein and N. S. Gingrich, Phys. Rev. **62**, 261 (1942).

The Forces Between Hydrogen Molecules The forces between neutral molecules are of three types: (1) attractive van der Waals, or polarization, forces; (2) forces due to multipoles (dipoles, quadrupoles, etc.) which may be repulsive or attractive, depending on the relative orientation of the molecules; (3) repulsive exchange forces which produce the "rigidity" of the structure and thus account for its shape and for its size. The first two types have previously been calculated for many different molecules, the last, because of its mathematical complexity, only for a few spherically symmetrical atoms.

In a recent publication, Margenau[1] has given a derivation of the exchange forces from the principles of quantum mechanics for the simplest molecule H_2. The calculation is based on a state function of the Heitler-London type, but with an adjustable value of the nuclear charge Z. The value of Z which minimizes the energy of a single molecule is 1.166, but it is found that this leads to quite erroneous consequences for the exchange forces. The source of this curious result is traced to the circumstance that in the bimolecular interaction problem the presence of *four* mutually repelling electrons produces an effective increase in the electron concentration about each nucleus, and hence an increase in Z. The choice finally adopted on the basis of this consideration is $Z=1.43$.

The magnitude of the exchange forces depends strongly on the relative orientation of the molecules. In the paper under review, the author gives results for three cases: (a) both molecular axes are parallel to the line joining the centers; (b) one axis is parallel, the other perpendicular; (c) both axes are perpendicular to that line. The intermolecular distances at which the forces set in decrease from case (a) to case (c), as the shape of the molecules suggests.

In order to obtain the total interaction, Margenau computes the quadrupole forces and the van der Waals forces, the latter with the use of an optical dispersion formula. The quadrupole forces are small except at distances of separation much greater than those of interest in connection with gas kinetic phenomena. When all three types are put together, the three curves given in Fig. 1 result; their labels refer to the relative orientations just specified. The ordinates represent interaction energies; the slopes are measures of the forces.

[1] H. Margenau, Phys. Rev. **64**, 131 (1943).

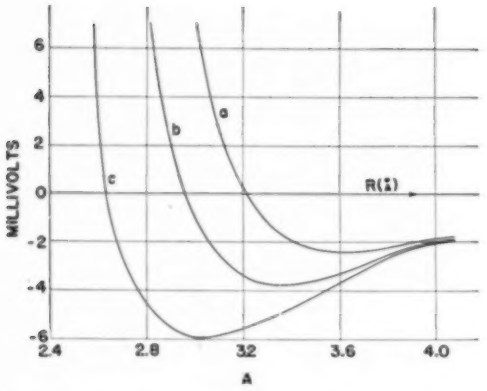


FIG. 1. Interaction energies when the approximately "right" value of $Z(Z=1.428)$ is used.

From observations of the second virial coefficient, Lennard-Jones and others have deduced empirical interaction curves for H_2 molecules. These do not, of course, permit a distinction between different orientations and are comparable to a mean of the three curves in Fig. 1. This interpretation leads to fairly satisfactory numerical agreement with the *a priori* results here discussed, for Lennard-Jones' curves have minima in the region from 3.3A to 3.5A and depths at minimum ranging from 2.7 to 2 millivolts.

New Books

Dynamical Analogies

BY HARRY F. OLSON. Pp. 196+xi, Figs. 12, 15×22½ cm. D. Van Nostrand Company, Inc., New York, 1943. Price $2.75.

Reasoning by analogy is a well-established practice in all engineering technique. It has been found particularly useful by those concerned with the dynamics of electrical circuits. Originally those engaged in this field found their knowledge of the movements of mechanical bodies of assistance in analyzing the behavior of new types of electrical circuits. Later, as these electrical circuits fostered the development of new forms of mechanical devices, such as acoustical reproducers, the process reversed and techniques developed for the analysis of electrical systems were used in the study of the behavior of mechanical systems. It is with those analytical techniques which may, with profit, be used both for electrical and for mechanical problems that Dr. Olson's book is concerned.

The book starts with a review of basic definitions and with a discussion of the fundamental elements of the several types of systems considered. These are the electrical, the mechanical rectilineal, the mechanical rotational, and the acoustical. The handling of dimensional units is interesting; conformity between the mechanical rectilineal and the electrical systems is obtained by the expedient of identifying self-inductance, electrical charge, and time, in the electrical case, with mass, linear displacement, and time, respectively, in the mechanical rectilineal case. This has the advantage of making all derived dimensional formulae in the two cases alike in form.

Tables for the intercomparison of the several fundamental and derived quantities are given. A table is included showing the dimensional formulae for all of the quantities in the several systems in their more familiar, or classical, form. In this latter table, by using electromagnetic units rather than electrostatic units, self-inductance has the dimensions of a length, hence the symbol L represents both self-inductance and length and serves as a bond between the conventional formulae and those showing the analogous relations.

Having established a working basis the treatment proceeds with the development of methods for the analysis of systems of one, two, and three degrees of freedom, working out the electrical, mechanical, and acoustical forms in each case. The usual difficulty associated with the parallel connection of two masses is met by the introduction of a differential gear train, in the case of rotational systems, or of a lever mechanism, in the case of rectilineal systems. Such artificial arrangements would suggest that the system were being fitted to the mathematics rather than the mathematics to the system.

The remainder of the book emphasizes the systems developed in the electrical field, namely, corrective networks, wave filters, and the transient phenomena therein. Chapters on driving systems and on generating systems are particularly useful since they indicate methods for expressing the effects of mechanical portions of a device as evidenced at the electrical terminals or vice versa. The previously established methods of mathematical correlation naturally tend to simplify such problems.

The book concludes with a group of applications, in the field of mechanics, which demonstrate the effectiveness of analytical techniques already well established in the electrical field.

J. WARREN HORTON
Columbia University, Division of War Research, New London, Connecticut

Tables of Functions

BY EUGENE JAHNKE AND FRITZ EMDE. Pp. 379, Figs. 181, 16½×24 cm. Dover Publications, New York, 1943. Price $3.50.

The reprinting of the *Tables of Functions* is timely and highly welcome. The tables have for over 30 years enjoyed great popularity among applied mathematicians, engineers, and physicists. They are useful as much for the numerical tables which they contain, as for the extensive formulae of the tabulated functions.

The bilingual text would seem to be an advantage, but

experience has shown that those familiar with both languages have to make a special effort to keep from "jay-reading" between the two columns. This reviewer would therefore prefer to see the future editions done either in English or in German.

C. L. PEKERIS
Columbia University
Division of War Research

Here and There

Staff Changes

Bert H. Norem has been appointed Assistant Professor of Administrative Engineering in the College of Applied Science, Syracuse University, Syracuse, New York.

On October 1, Cyril Tasker entered upon his duties as Director of Research of the American Society of Heating and Ventilating Engineers, 51 Madison Avenue, New York City. For the past 13 years, Mr. Tasker has been a member of the staff of the Ontario Research Foundation and as senior research fellow has carried on many important investigations relating to fuels, and a variety of problems connected with heating and air conditioning.

Save Electric Corporation, through Edward G. Kirby, President, announces the recent appointment of O. J. Schroeder, Vice President in Charge of Manufacturing, Equipment, and Engineering. Mr. Schroeder is part of the Corporation's expansion of Verd-A-Ray, its new lighting development. Among other new activities conducted by Mr. Schroeder will be a broad research program in physics and the physiological field as related to this new vitreous glass enamel coated lamp.

Bohr Reaches London

The *New York Times* reports that Dr. Niels H. D. Bohr, Danish physicist and Nobel Prize winner for atomic research, reached London by plane from Sweden on October 8, 1943.

Metropolitan Section of the American Physical Society

The Metropolitan Section of the American Physical Society will meet on February 25 and 26, 1944 at Brooklyn Polytechnic Institute. This meeting will be a Symposium on "Industrial Applications of X-Ray Diffraction" arranged by the American Institute of Physics.

Necrology

Professor Pieter Zeeman, noted scientist and winner of the Nobel Prize in physics in 1902, died in Amsterdam October 9, 1943, the *New York Times* reported. He was 78 years old. Professor Zeeman was known for his discovery of the "Zeeman effect," the phenomena produced in spectroscopy by the splitting up of spectral lines in a magnetic field. He was born in Zonnemaire, Zeeland, and studied at Leyden. In 1900 he was appointed Professor of Physics at Amsterdam and became Director of the Physical Institute in Amsterdam in 1908.

New Booklets

"Trail Blazers to Radionics and Reference Guide to Ultra-High Frequencies" is a booklet compiled by Miss Elizabeth Kelsey. It contains a bibliography of ultra-high frequency research as well as brief biographical sketches of great men of science who have contributed to the development of radionics. Requests for copies should be addressed to E. Kelsey, Zenith Radio Corporation, 680 North Michigan Avenue, Chicago 11, Illinois (152 pages).

"Permanent Magnet Manual No. 2" features the manufacture of permanent magnets and its varied applications. Copies are obtainable from the Indiana Steel Products Company, Valparaiso, Indiana (28 pages).

The Aerovox Research Worker is a monthly publication of the Aerovox Corporation. Its purpose is to bring to the radio experimenter and engineer authoritative first-hand information on condensers and resistances for radio work.

"Slidefilms and Motion Pictures to Help Instructors" is the title of a new and improved type of visual aids catalog-directory announced by The Jam Handy Organization, 2900 East Grand Boulevard, Detroit 11, Michigan. It will be sent free on request to any teacher, school, college, or educational group. By a new system of indexing, cross indexing and classifying, teaching slidefilms and motion pictures covering a wide range of studies, the teacher is enabled to locate quickly any subject needed. This catalog-directory is printed in colors, comprising 80 pages of detailed information—including the number of "frames" or pictures in each slidefilm and in each series. One special feature shows what projectors are best suited to various visualized teaching purposes.

Harry W. Dietert Company, 9330 Roselawn Avenue, Detroit 4, Michigan, is publishing a bi-monthly paper of interest to spectrographers and spectrographic laboratory personnel. The purpose of the paper is to disseminate technical information on spectrographic analyses and to acquaint interested personnel with latest developments. Those who make requests will be placed on a mailing list for future copies.

The Chemical Digest, a publication of Foster D. Snell, Inc., 305 Washington Street, Brooklyn, New York is published every few months and offers all sorts of chemical service.

Catalog 600 issued by the Precision Scientific Company, 1750 N. Springfield Avenue, Chicago 47, Illinois, is titled "Laboratory Utilities," comprising 112 pages, $8\frac{1}{2}'' \times 11''$, containing detailed information on an extensive variety of laboratory apparatus and equipment, many new "wrinkles" in familiar items, dozens of practical ideas for stepping up laboratory efficiency. Since all qualified laboratory supply dealers sell the apparatus shown in Catalog

600, it may be considered a timely supplement to dealer's catalogs. Laboratory utilities are defined as devices whose use can be adapted to virtually any type of laboratory in contrast to items of apparatus which would be restricted to specific procedures. Subjects include autoclaves, burners, centrifuges, digestion and extraction, distillation, heaters, hot plates, sieves, stirrers, shakers, titration illuminators, water stills.

"Portable Universal *p*H Indicator" catalog E-96(3), 1943 describes a new and improved universal *p*H indicator. This booklet can be obtained upon request from Leeds & Northrup Company, 4934 Stenton Avenue, Philadelphia 44, Pennsylvania (16 pages).

Innovations in Instruments

Allied Radio Receiver Kit

Allied Radio Corporation, Chicago, offers a modern radio receiver kit especially developed for illustrating theory and practices now being covered in basic or pre-induction radio training. It is now available to all schools and colleges conducting war training programs. This 5-tube kit permits progressive study of basic receiver subjects such as rectification, filtering, detection, r-f, i-f, and a-f amplifications, etc. It consists of all necessary parts, wire, hardware, solder, tubes, and speaker for the construction of a 5-tube a.c.-d.c. superhet receiver. Chassis is completely formed, punched, and rust-proofed. The only tools needed for assembly are screwdriver, pliers, and soldering iron. Circuit includes many outstanding features such as oscillator biased r-f and i-f stages, contact-potential biased audio stage, inverse feedback, automatic volume control, high capacity filtering, self-contained antenna, and others. Free descriptive material and circuit diagrams can be obtained by request from the Allied Radio Corporation, Educational Division, 833 West Jackson Boulevard, Chicago 7, Illinois.

Ross-Adams Lens Tissues

The Ross-Adams Lens Tissues, distributed by Clay-Adams Company, of New York City, are fast becoming popular. They represent a radical departure from the usual Japanese style lens tissues. These tissues are specially processed, dense, and have considerable tensile strength. They will not disintegrate with manipulation. The surface is smooth, thereby tending to prevent abrasive dust particles from becoming embedded in the paper and they have an unusual grease-absorbing quality, easily removing fingermarks and other oils and greases from lenses. They are smooth, dense, and thin as compared with the soft, porous, Japanese style lens tissues.

Recent Applications of Physics

Powder Metallurgy for Magnets

Using powdered iron, P. R. Kalischer, metallurgist at the Westinghouse Electric and Manufacturing Company, has been fashioning small magnets for electrical circuit breakers—automatic switches controlling the flow of power in war plants and aboard naval vessels.

The process is said to resemble closely the art of brick-making. Bricks are fashioned by pressing clay in a mold; magnets are formed from iron powder by pressing the powder in a finely-machined die. Bricks must be baked in a kiln to harden; the magnets are sintered or fired in a furnace with a hydrogen atmosphere, preventing oxygen from reaching the metal and forming an oxide coating on the magnets.

To make these magnets Mr. Kalischer fits a plunger into the powder-filled die and places both plunger and die in a hydraulic press. As the plunger is pushed down into the die cavity, the particles of metal are squeezed closer and closer together until finally each one is welded to its neighbors. It is then fired in a furnace kept at a temperature far below the melting point of iron but high enough to force metal atoms to travel from one particle to another, knitting the whole structure tightly together. Powdered metal parts can be pressed to within a thousandth of an inch of the size desired. This reduces tedious machining and saves scrap.

A variation of this trick of making tough metals from powders makes possible the low cost and long-lived electric lamps now in use. Tungsten ore, a coarse brown powder, is chemically treated to convert it into yellow tungsten oxide, also a powder. Then the powder is squeezed with pressures of 20 tons per square inch to form it into a two-foot long bar less than half an inch in thickness, which is baked in a hydrogen furnace. Later it is placed in a hydrogen-filled metal "bottle" while an electric current is passed through it. The next step is to hammer the bars into rods which can be drawn into wires one-fifth the thickness of human hair. This material is stronger than the highest grade of steel

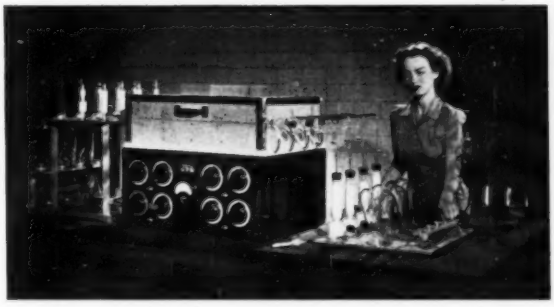
Multiple Unit Electrically Heated Combustion Furnace

Incident to the stepped-up production of high octane aviation gasoline essential to the war program, the Universal Oil Products Company of Chicago, Illinois, has developed a multiple unit electrically heated combustion furnace offering several novel features. The equipment is manufactured and offered commercially by the Precision Scientific Company, 1750 N. Springfield Avenue, Chicago, Illinois, through cooperation with the Universal Oil Products Company.

Although originally designed specifically for the determination of carbon on cracking catalysts used for producing high octane aviation gasoline, the equipment is modifiable to handle a wide variety of organic combustions within

the temperature limit of 540°C (1000°F). Briefly, the equipment comprises: (1) Oxygen purification system for combustion gases. (2) Oxygen pressure regulating column. (3) Electrically heated furnace 24″ long, with four combustion tubes 1″ diameter×30″ long. Heating intensity for each half of each tube is independently controlled, and temperature of any tube can be readily observed by means of an indicating pyrometer. (4) Conventional absorption trains with calcium chloride for absorption of moisture and Ascarite for removal of carbon dioxide.

The assembly illustrated is merely representative of other multiple assemblies, which can be built up to suit the job involved.



Magnetic Optical System Setting up and adjusting a standard optical system has been considerably simplified by the insertion of Alnico magnets into the base of the standards. This form of optical system was devised by Norman F. Barnes of General Electric's laboratory in Schenectady. All that is required is a metal top to the table on which the system is placed and metal walls, if a vertical system is desired. The magnets are sufficiently strong to hold the parts in correct alignment.

Mass Production X-Ray Unit A new mass production x-ray machine capable of inspecting as many as 17,000 airplane castings in a 24-hour day has been developed by Westinghouse for a midwestern war plant. This brings the advantages of assembly line speed to x-ray work and makes it possible to x-ray metal castings for defects at a rate of one every five seconds.

The unit uses a moving conveyor 40 feet long and three feet wide that transports the castings through the x-ray inspection and provides the fastest method yet devised to spot flaws in large quantities of metal parts. It produces an exposed film of six castings every 30 seconds to provide an almost continuous flow of exposed film ready for developing. When developed, each film shows an inside view of the six castings. Faulty castings with blow holes, cracks, and other defects then can be weeded out. Designed to inspect both engine and fuselage castings up to five inches in thickness, the mechanism has been installed at a Detroit, Michigan, plant of a large automobile manufacturer producing war materials.

Inside two steel towers, each 12 feet high and situated near the middle of the conveyor, the actual x-ray inspection is conducted. Each tower houses an x-ray tube—one tube operating at 140,000 volts, the other at 220,000 volts—so that castings of different types and thicknesses can be examined at the same time.

The castings move along the conveyor on trays that halt automatically under the x-ray tubes. A lead-lined protective device shields workmen from x-radiation while the exposure is made, and the castings then continue along the conveyor. For quickly identifying any defective castings, corresponding numbers are given the film and the castings. The unit can be operated by one man or eight men, depending upon the volume and speed of production desired. Owing to the safety factor provided by the lead-lined hoods, the unit can be located anywhere in the plant and other work can continue without interruption while the x-ray exposures are being made. Previous methods required a special lead-lined room which had to be temporarily evacuated during exposures, resulting in loss of time and efficiency. Although it will be used chiefly to test aluminum castings, the machine can inspect other metals also.

Motors for Stratosphere Wind Tunnel Construction of two 20,000-horsepower electric motors for the Army's new 600-mile-per-hour stratosphere wind tunnel at Wright Field has been started by the Westinghouse Electric and Manufacturing Company. The new tunnel will be a rectangle of giant metal pipes 600 feet long and varying in diameter from 10 feet to 40 feet. It will be built close to the two-year-old, 400-mile-an-hour tunnel which is powered by a single 40,000-horsepower Westinghouse motor, largest of its kind in the world. The test chamber of the new tunnel will duplicate the conditions pilots and planes must cope with in the lower stratosphere, seven or eight miles above earth. In this cloudless region the temperature is constantly 67 degrees below zero and the air pressure is less than one-fifth as great as it is at sea level.

The two propellers will produce a smooth, non-turbulent flow of air. The two motors with top speeds of 465 r.p.m. will be mounted at adjacent corners of the tunnel and each will be connected to its propeller by a 40-foot long shaft piercing the tunnel wall. The motors can be slowed down to 50 r.p.m. reducing the hurricane in the tunnel to a moderately stiff wind. A blower under each motor will send 50,000 cubic feet of air a minute over the cores and windings to carry away the motor's heat. Each motor will be about 11 feet high and 25 feet long, including shaft and bearings.

So heavy is the rotor, or revolving central part of the motor—33 tons—that if left at rest for several days, it would squeeze the film of oil out of the space between the motor shaft and the bearings supporting it. When this happens a pumping system will force oil into this space, lifting the massive rotor several thousandths of an inch so the shaft will not rub directly on its supporting bearing.

Contributed Original Research

Theory of Filler Reinforcement in Natural and Synthetic Rubber. The Stresses in and about the Particles

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(Received August 7, 1943)

An attempt is made to develop a general theory of filler reinforcement by determining the stresses occurring in and about a spherical particle imbedded in a rubberlike medium subjected to an applied tension. For a system containing a single particle rigidly attached to the adjacent medium, an application of the theory of elasticity shows that, for infinitesimal deformations, the stress components within the particle are independent of the radial distance from the origin, taken at the center of the particle. The stress components at a given point in the surrounding medium depend on the elastic constants both of the particle and of the medium, on the radius of the sphere, on the distance from the origin, and on the angle between the direction vector and the applied tension. Expressions are given for the average stresses in media containing many (independent) particles. Theoretical values of the bulk moduli of the synthetic rubbers considered in the treatment are derived from sound velocity data. Curves showing the spatial distribution of radial and shear stresses are presented for a range of values of elastic constants to be expected for different kinds of filler particles and rubberlike materials.

THE mechanism by which finely divided fillers, notably carbon black, give rise to greatly improved mechanical properties of rubber compounds has been the object of many investigations. The extensive literature on this subject has been reviewed rather recently by Hock,[1] and also by Shepard, Street, and Park.[2] The great majority of that work and the theories derived therefrom are based on results obtained with natural rubber. Briefly stated, the two factors most generally invoked in order to explain filler reinforcement are (at least for more or less spherical particles) (1) the specific surface area of the particles, and (2) the strength of the bond, or interfacial energy, at the filler-rubber boundary. Yet, it is a matter of common experience that when an effort is made to apply such information or theory to synthetic rubbers, one encounters irregularities that are little understood and are often attributed to factor (2). It seems reasonable to expect that a satisfactory theory of filler reinforcement should be equally applicable not only to fillers of varying specific surface area and composition, but also to a variety of natural and synthetic rubbers. Furthermore, while one can hardly deny that the strength of the filler-rubber interface is an important factor in reinforcement behavior, the older theories do not make it evident how this bonding brings about reinforcement. It is therefore desirable to develop a general theory of filler reinforcement that has greater mechanistic clarity.

In a recent attempt of this sort, Weiss[3] considered the total increase in free energy due to filler to consist of two parts, (a) the total surface free energy F_s of the particles in the mixture, and (b) the total elastic energy F_e set up in the matrix by the filler. In common with earlier workers[1, 2] he

[1] L. Hock, in K. Memmler, *Science of Rubber* (Reinhold Publishing Corporation, New York, 1934), pp. 495 *et seq.*
[2] N. A. Shepard, J. N. Street, and C. R. Park, in C. C. Davis and J. T. Blake, *Chemistry and Technology of Rubber* (Reinhold Publishing Corporation, New York, 1937), pp. 380 *et seq.*
[3] J. Weiss, Trans. Inst. Rubber Ind. **18**, 32 (1942); Rubber. Chem. Tech. **16**, 124 (1943).

suggested that information on F_s might be obtained by measuring the heat of wetting of fillers by organic liquids. In order to give expression to the elastic energy contribution, Weiss applied a theory developed by Mott and Nabarro[4] for the case of precipitation hardening in metals. The physical concept involved in his treatment is that a particle of filler imbedded in rubber will generally have a different volume from the actual space (or "hole") in the material available for it. If it is held firmly enough by the surface forces, the particle will compress or expand the surrounding medium, depending on whether it is larger or smaller than the "hole" in the matrix, and thus set up a more or less symmetrical stress field. Although Weiss' treatment is valuable in emphasizing the necessity for considering the stress field, it has the disadvantages of introducing a rather arbitrary "hole" parameter, and, what is more important from the standpoint of rubber behavior, it does not show how the internal stresses depend on the degree of deformation or on the magnitude of an applied force. In a treatment independently worked out by the writer, and presented below, an attempt is made to develop this subject and to point out its bearing on the general problem of reinforcement.

THE STRESSES NEAR SPHERICAL INCLUSIONS IN ELASTIC MEDIA

The problem here considered is that of determining the stresses in the neighborhood of a spherical particle of radius a imbedded in an elastic medium of infinite extent, the latter being subjected to a uniform tension T at infinity. It is assumed that the particle is rigidly attached to the adjacent layer of the elastic medium, and that the latter is subjected only to infinitesimal deformations. It is then possible to apply the results of the classical theory of elasticity. The general equations of equilibrium of an elastic body,[5] expressed in spherical coordinates and omitting the azimuthal component of motion, may be written

$$(\lambda+2\mu)\frac{\partial\Delta}{\partial r}-(2\mu/r)\frac{\partial\tilde{\omega}}{\partial\theta}-(2\mu/r)\tilde{\omega}\cot\theta=0,$$
$$[(\lambda+2\mu)/r]\frac{\partial\Delta}{\partial\theta}+2\mu\frac{\partial\tilde{\omega}}{\partial r}+2\mu\tilde{\omega}/r=0, \tag{1}$$

where λ and μ are Lamé's elastic constants, u and v are the radial and colatitudinal components of displacement, and

$$\Delta=\frac{\partial u}{\partial r}+2\mu/r+(1/r)\frac{\partial v}{\partial\theta}+(v/r)\cot\theta,$$
$$2\tilde{\omega}=\frac{\partial v}{\partial r}+v/r-(1/r)\frac{\partial u}{\partial\theta}. \tag{2}$$

These equations were solved for the case of an imbedded spherical particle by Sezawa and Miyazaki[6] to a degree of approximation including the second order of spherical harmonics, omitting terms of polar unsymmetry and terms which would make the stresses at infinity very large. Since their calculations appear to have been completely overlooked, probably because of general inaccessibility, the complete results of their solution are given below. Denoting the elastic constants of the particle by accents, those of the surrounding medium being unaccented, the stress components within the sphere were found to be

$$p_{rr}'/T=(\lambda+2\mu)(3\lambda'+2\mu')/(3\lambda+2\mu)(3\lambda'+2\mu'+4\mu)+(4\mu'D_1/D)P_2(\cos\theta), \tag{3a}$$

$$p_{\theta\theta}'/T=(\lambda+2\mu)(3\lambda'+2\mu')/(3\lambda+2\mu)(3\lambda'+2\mu'+4\mu)+2\mu'D_1/D-(4\mu'D_1/D)P_2(\cos\theta), \tag{3b}$$

$$p_{\phi\phi}'/T=(\lambda+2\mu)(3\lambda'+2\mu')/(3\lambda+2\mu)(3\lambda'+2\mu'+4\mu)-2\mu'D_1/D, \tag{3c}$$

$$p_{r\theta}'/T=(2\mu D_1/D)\frac{dP_2(\cos\theta)}{d\theta}. \tag{3d}$$

[4] N. F. Mott and F. R. M. Nabarro, Proc. Phys. Soc. London **52**, 86 (1940).

[5] A. E. H. Love, *The Mathematical Theory of Elasticity* (Cambridge University Press, London, 1927), fourth edition, p. 141.

[6] K. Sezawa and B. Miyazaki, J. Soc. Mech. Eng. Tokyo **31**, 625 (1928).

The stresses in the medium are given by the relations

$$p_{rr}/T = 1/3 + \{4\mu[3(\lambda'-\lambda)+2(\mu'-\mu)]/(3\lambda+2\mu)(9\lambda'+6\mu'+12\mu)\}(a^3/r^3) + \{2/3+[(9\lambda+10\mu)D_2/3D](a^3/r^3) + (24\mu D_3/D)(a^5/r^5)\}P_2(\cos\theta), \quad (4a)$$

$$p_{\theta\theta}/T = 2/3 - \{2\mu[3(\lambda'-\lambda)+2(\mu'-\mu)]/(3\lambda+2\mu)(9\lambda'+6\mu'+12\mu)+\mu D_2/3D\} \times (a^3/r^3)+(2\mu D_3/D)(a^5/r^5) - \{2/3+(\mu D_2/3D)(a^3/r^3) + (14\mu D_3/D)(a^5/r^5)\}P_2(\cos\theta), \quad (4b)$$

$$p_{\phi\phi}/T = \{\mu D_2/3D - 2\mu[3(\lambda'-\lambda)+2(\mu'-\mu)]/(3\lambda+2\mu)(9\lambda'+6\mu'+12\mu)\}(a^3/r^3) - (2\mu D_3/D)(a^5/r^5) - \{(\mu D_2/D)(a^3/r^3) + (10\mu D_3/D)(a^5/r^5)\}P_2(\cos\theta), \quad (4c)$$

$$p_{r\theta}/T = \{1/3 - [(3\lambda+2\mu)D_2/6D](a^3/r^3) - (8\mu D_3/D)(a^5/r^5)\}\frac{dP_2(\cos\theta)}{d\theta}, \quad (4d)$$

where $P_2(\cos\theta)$ is the Legendre function of the second degree, and the D's are functions of the elastic constants, and are given by the determinants

$$D = \begin{vmatrix} \lambda'/7 & 4\mu' & -(9\lambda+10\mu)/3 & -24\mu \\ -(8\lambda'+7\mu')/21 & 2\mu' & (3\lambda+2\mu)/6 & 8\mu \\ -\lambda'/7\mu' & 2 & (3\lambda+5\mu)/6\mu & 3 \\ -(5\lambda'+7\mu')/42\mu' & 1 & 1/6 & -1 \end{vmatrix}, \quad (5a)$$

$$D_1 = \begin{vmatrix} \lambda'/7 & 2/3 & -(9\lambda+10\mu)/3 & -24\mu \\ -(8\lambda'+7\mu')/21 & 1/3 & (3\lambda+2\mu)/6 & 8\mu \\ -\lambda'/7\mu' & 1/3\mu & (3\lambda+5\mu)/6\mu & 3 \\ -(5\lambda'+7\mu')/42\mu' & 1/6\mu & 1/6 & -1 \end{vmatrix}, \quad (5b)$$

$$D_2 = \begin{vmatrix} \lambda'/7 & 4\mu' & 2/3 & -24\mu \\ -(8\lambda'+7\mu')/21 & 2\mu' & 1/3 & 8\mu \\ -\lambda'/7\mu' & 2 & 1/3\mu & 3 \\ -(5\lambda'+7\mu')/42\mu' & 1 & 1/6\mu & -1 \end{vmatrix}, \quad (5c)$$

$$D_3 = \begin{vmatrix} \lambda'/7 & 4\mu' & -(9\lambda+10\mu)/3 & 2/3 \\ -(8\lambda'+7\mu')/21 & 2\mu' & (3\lambda+2\mu)/6 & 1/3 \\ -\lambda'/7\mu' & 2 & (3\lambda+5\mu)/6\mu & 1/3\mu \\ -(5\lambda'+7\mu')/42\mu' & 1 & 1/6 & 1/6\mu \end{vmatrix}. \quad (5d)$$

It can be seen from Eqs. (3) that the stress components within the sphere are independent of the radial distance. The stresses at a given point in the surrounding medium depend on the elastic constants both of the sphere and of the medium, as shown by Eqs. (4) and (5), on the radius a of the sphere, on the distance r from the center of the sphere to the point under consideration, and on the angle θ that the latter line makes with the direction of the externally applied traction (see Fig. 1).

AVERAGE STRESSES DUE TO MANY INDEPENDENT PARTICLES

In order to make a closer examination of the stress relations, let us consider the radial stress p_{rr} in the medium at a point lying on the direction

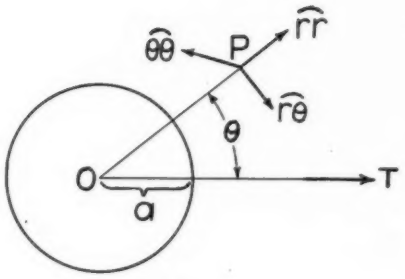

FIG. 1.

vector of the applied tension. Then $\theta=0°$, and Eq. (4a) may be written

$$p_{rr}/T=c_1+c_2(a^3/r^3)+c_3(a^5/r^5), \qquad (6)$$

where c_2 and c_3 are functions of the elastic constants. If there are N similar particles in unit volume, and if these be sufficiently far apart to avoid the effects of particle interactions (which will not be the case for, say, a tire tread composition, in which the mean particle separation is comparable in magnitude to the particle diameter), then the average distance $\bar{r}$ between the centers of the particles is of the order $(1/N)^{\frac{1}{3}}$, and the average radial stress, according to Eq. (6), becomes

$$(p_{rr}/T)_{Av}=c_1+c_2'Na^3+c_3'(Na^3)^{5/3}. \qquad (7a)$$

Since the volume fraction v_f of the filler present is proportional to Na^3, the average stress may be written

$$(p_{rr}/T)_{Av}=c_1+c_2''v_f+c_3''v_f^{5/3}. \qquad (7b)$$

The first term in Eq. (7b) is obviously the stress that would be obtained when tension is applied to an elastic medium containing no filler particles ($v_f=0$), while the remaining terms represent the filler contribution. The average stress is found to have a maximum at $v_f=(-3c_2''/5c_3'')^{\frac{1}{2}}$. In this connection it may be worth while to point out that, as is commonly known, the reinforcing properties of active fillers do not simply increase with increasing filler concentration, but pass through a maximum whose location depends primarily on the nature of the filler (for a given kind of rubber). (It is hardly necessary to point out that Eq. (7b) cannot be used to determine *quantitatively* the optimum filler concentration since the latter generally occurs for such high values of N that the effects of particle interactions are, in all probability, not negligible.) The average elastic energy associated with the radial stress is proportional to $(p_{rr}/T)^2_{Av}$ and likewise has a maximum at the same value of v_f as does the average stress.

For a given direction θ (other than $\theta=0°$) it is seen that the shear stress, Eq. (4d), has the same form as Eq. (6) (but with different values of the coefficients). The average shear stress for a system containing many particles is therefore given by a relationship similar to Eq. (7b), but with a different value of the filler concentration at the maximum. In other words, the filler concentration required to give a maximum value to the average tensile stress will not, in general, coincide with that giving the maximum average shear stress.

The average radial (and likewise shear) stress is seen from Eq. (7b) to be independent of the size of the particles, but depends only on their concentration, and on the values of the elastic constants represented by the coefficients.

APPLICATION TO NATURAL AND SYNTHETIC RUBBERS

Since the stresses in a system containing a single particle would be expected to give at least a semiquantitative insight into the mechanical behavior of many-particle systems in which the particles are not sufficiently far apart to avoid interaction, it is of interest to determine the radial dependence of the stress-components of Eqs. (4). Since this evaluation requires a knowledge of the elastic constants of both phases, it is unfortunate that scarcely any such data are to be found in the literature for materials used as rubber fillers, and none for synthetic rubbers.

Carbon blacks, for which no elastic constants are available, are the fillers of greatest value and interest. The bulk modulus K for graphite, according to Richards,[7] is 4.9×10^6 lb./sq. in., presumably at room temperature. It is assumed here that the values of K for carbon blacks do not differ much from that of graphite. In view of the graphite-like nature of carbon blacks,[8] this as-

[7] T. W. Richards, J. Am. Chem. Soc. **37**, 1643 (1915).
[8] J. Biscoe and B. E. Warren, J. App. Phys. **13**, 364 (1942).

sumption seems to be reasonably good for the types of carbon blacks most commonly used as rubber fillers, but would doubtless be inadmissible for some types, the particles of which appear to have a considerable amount of porosity.[9] Furthermore, results based on this value of K should be qualitatively applicable to other kinds of fillers since the bulk moduli of most solids are of the same order of magnitude[10] (Table I). The modulus

TABLE I. Cubical compressibilities of some solids.

Material	Compressibility (cm^2/kg) $\times 10^6$
Graphite	3.0
Aluminum oxide (sapphire)	0.317
Magnesium oxide (pressed powder)	0.986
Other oxides, crystalline salts, sugars, and minerals of widely different structures and compositions	0.3–19

of rigidity μ of any isotropic solid is related to K and to the Poisson ratio σ by the equation $\mu=3K(1-2\sigma)/2(1+\sigma)$. Since σ for graphite is not given in the literature, we assume it to have a value in the normal range of 0.25–0.33. The rigidities corresponding to these limits are 2.9×10^6 and 1.8×10^6 lb./sq. in., respectively. It will be seen later that the resulting stresses are not appreciably sensitive to the variation thus introduced by the uncertainty in σ. Since, for an isotropic solid, $K=\lambda+2\mu/3$, the corresponding values of λ are 3.0×10^6 and 3.7×10^6 lb./sq. in. In order to simplify the numerical calculations, rounded values of $\lambda=4\times10^6$ and $\mu=2\times10^6$ will be used for graphite.

From the work of Adams and Gibson[11] it is found that the value of K for vulcanized natural rubber is about 4×10^5 lb./sq. in. at pressures in the vicinity of one atmosphere. Since a tire tread composition will have a modulus of rigidity of roughly 100 lb./sq. in.[12,13] we may use $\lambda=4\times10^5$ and $\mu=100$ as typical values for the rubber medium.

[9] W. R. Smith, private communication.
[10] Landolt-Börnstein, *Physikalisch-chemische Tabellen* (Verlag Springer, Berlin, 1935), third supplement, I, 69.
[11] L. H. Adams and R. E. Gibson, J. Wash. Acad. Sci. **20**, 213 (1930).
[12] H. Hencky, Rubber. Chem. Tech. **6**, 217 (1933).
[13] M. Mooney, J. App. Phys. **11**, 582 (1940).

The curves in Fig. 2 show the effects of varying the values of the elastic constants on the radial

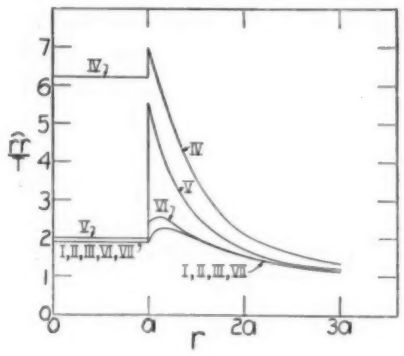

FIG. 2. Calculated radial stress p_{rr} in and about a spherical particle as a function of distance and of the elastic constants for $\theta=0°$. The curves are numbered in accordance with the data given in Table II.

stress at various distances from the imbedded particle along the direction of the applied tension $T(\theta=0°)$. These curves were calculated by means of Eqs. (3a) and (4a), the elastic constants used for this purpose being tabulated, together with the corresponding values of the various determinants [Eqs. (5)], in Table II. The stress is

TABLE II. Elastic constants and corresponding determinants.

Curve	λ' $\times10^{-6}$	μ' $\times10^{-6}$	λ $\times10^{-5}$	μ	$-D$ $\times10^{-15}$	$-D_1$ $\times10^{-11}$	$-D_2$ $\times10^{-11}$	D_3 $\times10^{-13}$
I	4	2	4	100	39.6	0.826	1.65	6.60
II	3	3	4	100	56.6	0.786	2.36	9.43
III	4	2	6	100	59.4	1.24	1.65	9.90
IV	4	2	6	50	59.4	4.38	3.30	19.8
V	1	0.5	4	100	2.48	0.206	0.103	0.0476
VI	1	0.5	4	50	4.67	0.370	0.195	1.56
VII	4	2	4	50	79.2	1.65	3.30	26.4

observed to be independent of distance within the particle ($r<a$). At the surface, the stress in the medium rises discontinuously and rapidly to a maximum, the position of which depends on the set of constants chosen, and then decreases asymptotically to the value of the applied tension. For a particle such as graphite* in natural rubber, a comparison of curves I and VII shows that a twofold change in the rigidity of the medium does not affect the stress. Curves I and III show that, for $\mu=100$, a 50 percent increase in the bulk modulus of the medium likewise has no appreci-

* Or, more strictly speaking, a spherical particle having the mean elastic properties of graphite.

able effect on the stress. However, if this particle be imbedded in a medium the bulk modulus of which is 50 percent greater than that of natural rubber, it is seen from curves III and IV that a twofold change in the rigidity of the medium has a large effect on the stress, the latter being more than three times as large, at the surface of the particle, for the medium having the lower rigidity.

A comparison of curves I and II shows that the uncertainty in the value of the Poisson ratio for graphite is not reflected in any change in the stress.

If the graphite particle is replaced by one the compressibility of which is four times as large, it is observed (curve V) that the stress at the surface of the particle is increased almost threefold when $\mu=100$ for the medium. If, on the other hand, $\mu=50$, the increase in stress (curve VI) is much less.

Figure 3 shows the behavior of the shear stress

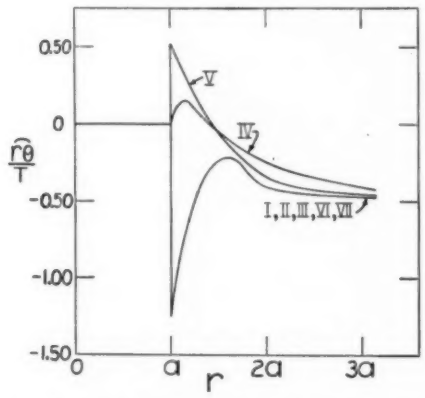

FIG. 3. Calculated shear stress $p_{r\theta}$ in and about a spherical particle as a function of distance and of the elastic constants for $\theta=45°$. The curves are numbered in accordance with the data given in Table II.

$p_{r\theta}$ as a function of the elastic constants. The curves were calculated by means of Eqs. (3d) and (4d) with the sets of constants given in Table II, for points on a line making an angle of $\theta=45°$ with the direction of applied tension. It is seen that, as in the case of the radial stress, the values of the elastic constants have a considerable influence on the shear stress. Since the general comparisons are very similar to those made above for the case of the radial stress, no more detailed discussion will be made for this case.

It may be concluded that, in order to obtain a state of minimum stress, it is necessary to obtain a proper balance, so to speak, between the elastic constants of the imbedded particle and of the medium. The curves indicate that a series of particles possessing different values of elastic constants will not necessarily show the same order of behavior when the medium in which they are imbedded is changed. It is therefore of interest to consider to what extent the elastic constants of various rubberlike materials differ.

ELASTIC CONSTANTS OF SYNTHETIC RUBBERS

In order to make quantitative estimates of the stresses, a knowledge is required of the values of the elastic constants of both the filler and the rubber medium. The extent of variation to be expected in the case of fillers has already been considered. The maximum variation in the compressibility of solids, as shown in Table I, is about fifty-fold, and the corresponding bulk moduli will have a spread of the same order. No data are available for the elastic constants of synthetic rubbers. For the purpose of estimating the range of values that such materials may be expected to exhibit, it is therefore necessary to resort to theoretical calculations.

The molecular sound velocity V is defined by the relation

$$v_s=(Vd/M)^3, \tag{8}$$

where v_s is the velocity of sound, d is the density, and M is the molecular weight. It has been shown[14,15] that, for a large number of different kinds of organic liquids, V is an accurately additive function of what have been termed "bond velocities." The latter have been determined[15] for various chemical linkages. With the aid of these values, and one supplementary bond velocity[16] that was not determined by Lagemann and Corry, it is possible to calculate by means of Eq. (8) the velocity of sound in the synthetic rubbers listed in Table III, and from these

[14] M. R. Rao, J. Chem. Phys. **9**, 682 (1941).
[15] R. T. Lagemann and J. E. Corry, J. Chem. Phys. **10**, 759 (1942).
[16] L. Bergmann, *Ultrasonics* (Bell and Sons Ltd., London, 1938), p. 122, gives $v_s=1275$ m/sec. for acetonitrile at 28°C ($d=0.775$). By means of Eq. (8) and the known (reference 15) contributions of the C—H and C—C bonds, we obtain a bond velocity of 284 for the —C≡N group.

TABLE III. Calculated compressibility data for synthetic rubbers.

Rubber	d	v_s(m/sec.)	Bulk modulus (lb./sq. in.) $\times 10^{-5}$
Polychloroprene	1.25	1720	5.44
Methyl rubber	0.93	1930	5.07
Polyisobutylene (or Butyl rubber)	0.92	1930	4.90
Perbunan*	0.96	1790	4.54
Buna S**	0.94	1690	3.92
Polyisoprene	0.9	1710	3.87
Polybutadiene	0.9	1650	3.59

* Calculated for a ratio of three moles of butadiene to one mole of acrylonitrile.
** Calculated for a ratio of five moles of butadiene to one mole of styrene.

results the isothermal[17] compressibilities and bulk moduli. The calculated value for polyisoprene (3.87×10^5) is in surprisingly good agreement with the experimental value (3.98×10^5) obtained by Adams and Gibson[11] for vulcanized natural rubber. Since no other experimental values are available for comparison with the remaining data of Table III, it cannot be stated with certainty whether or not this agreement for natural rubber is accidental. Since the modulus of rigidity is not a characteristic constant, but may be altered over rather wide limits by the use of various compounding ingredients and conditions of vulcanization, no values are listed for this parameter. For the synthetic rubbers considered here, it is seen that the extreme values of the bulk modulus (and therefore λ) differ by roughly 50 percent. It was for this reason that the range of values for λ given in Table II was selected for the stress calculations.

DISCUSSION

The treatment presented in this paper serves to show that the stresses occurring in the vicinity of particles imbedded in rubberlike media depend on the elastic constants of both phases. It should be emphasized, however, that any attempt to apply these results in a quantitative sense to the problem of predicting conventional "moduli" or tensile strengths of rubber containing fillers is not justified for the following reasons. Derivations based on the mathematical theory of elasticity assume that the conditions of infinitesimal deformations, conformity with Hooke's law, and perfect elasticity are met. It is obvious that the usual deformations involved in the determination of tensile properties, and the common occurrence of hysteresis and plastic flow, do not fall within the scope of the theory.[18] Furthermore, it was necessary, in order to obtain mathematical solutions, to assume that the elastic medium is rigidly bound to the surface of the particle, and that the latter is spherical. The assumption of rigid binding is certainly not true for all fillers, although it appears to be valid for carbon blacks and possibly for other fillers with outstanding reinforcing characteristics.[19] For technical mixtures composed of high concentrations of filler particles with resulting small distances of particle separation, the theoretical considerations are further complicated by particle interactions.

Photoelasticity studies carried out with a large disk of hard rubber cemented into a soft rubber sheet[20] show very good agreement between the observed stresses and those calculated[21] for the two-dimensional problem, and support the belief that the agreement for the three-dimensional problem would be equally good if it were possible to carry out the experiments for this case. Barnett[22] studied the stresses in a soft rubber sheet containing a large hard rubber inclusion by observing the distortion produced in a pattern marked on the sheet, and concluded that the stresses are greater at the surface of large particles

[17] From the velocity of sound one obtains the adiabatic compressibility by means of the relation $v_s=(1/d\beta_{ad})^{\frac{1}{2}}$. The isothermal compressibility follows from the thermodynamic relationship $\beta_{iso}=\beta_{ad}+\alpha^2T/dc_p$ where α is the expansivity, T the absolute temperature, and c_p the specific heat at constant pressure. At ordinary temperatures, and for materials whose values of α and c_p are of the same order of magnitude as those of natural rubber, the second term is negligible.

[18] Nevertheless, one can by various practical techniques eliminate, or at least minimize, the latter two phenomena. It may be possible to treat the stress problem in the region where divergence from Hooke's law occurs by utilizing the theory of large deformations developed by Mooney (reference 13).

[19] H. F. Schippel, Ind. Eng. Chem. **12**, 33 (1920); H. Green, *ibid.* **13**, 1029 (1921); H. A. Depew and M. K. Easley, *ibid.* **26**, 1187 (1934); F. Boiry, Rev. gén. Caoutchouc **3**, No. 24, 12; No. 25, 11 (1926).

[20] W. E. Thibodeau and L. A. Wood, J. Research Nat. Bur. Stand. **20**, 393 (1938).

[21] K. Sezawa and G. Nishimura, Rep. Aeron. Res. Inst. Tokyo Imp. Univ. **6**, 25 (1931).

[22] C. E. Barnett, Physics **7**, 189 (1936).

than smaller ones. This conclusion is at variance with the results of the calculations given in this paper for spherical inclusions.

In addition to their bringing about photoelastic effects and pattern distortions, the stresses are revealed by their influence on the shape of the spots obtained in x-ray diffraction diagrams of rubber-filler systems. Gehman and Field[23] have made such measurements and found this to be the most satisfactory interpretation of their results. According to their explanation, the distortion of the lines of stress in the neighborhood of a particle results in a fiber axis making a varying angle with the direction of the applied tension, this orientation causing the x-ray spots to broaden into the observed arcs. Their observation that the crystallites are less perfectly aligned along the axis of stretch for channel black loadings as compared with other blacks of larger particle size may be attributable, according to the theory herein presented, either to the differences in the elastic constants of the different types of filler particles, or perhaps to different states of cure with resulting differences in the moduli of rigidity.

One would expect the stresses to vary with temperature; however, the magnitude of this effect cannot be estimated at present, since it would be required to know the temperature dependence of the elastic constants. Also, qualitative predictions are complicated by the fact that, while the bulk modulus and rigidity of the solid particle decrease with increasing temperature, the Young's modulus (or rigidity) of the natural or synthetic rubbers increases with temperature,[24] and a compensating tendency may thus occur. Furthermore, at low temperatures crystallization in the rubber medium would also have to be considered; first, as modifying the average elastic constants of the medium, and second, as producing in effect further heterogeneity through the formation of crystallite "particles" imbedded in the otherwise amorphous medium.

ACKNOWLEDGMENT

The author takes this opportunity to express his appreciation to his colleague, Dr. P. J. Flory, who read the manuscript and offered valuable suggestions.

[23] S. D. Gehman and J. E. Field, Ind. Eng. Chem. **32**, 1401 (1940).

[24] R. L. Anthony, R. H. Caston, and E. Guth, J. Phys. Chem. **46**, 826 (1942); L. E. Peterson, R. L. Anthony, and E. Guth, Ind. Eng. Chem. **34**, 1349 (1942).

A Treatment of Non-Linear Devices Based upon the Theory of Related Linear Functions

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Because of the mathematical difficulties involved in the treatment of large-signal detectors, experimental methods for obtaining design data were early adopted. The procedure has been to replace the detector by a "model" described by a rectification or transrectification diagram. The "model" for a large-signal detector is developed from practical measurement results. Formulae and results may be extracted from the "model" if the existing analogy with the plate-current *vs.* plate-voltage diagram for a vacuum tube is made use of. This fundamental procedure may be extended to other non-linear devices, and in particular to frequency converters. It is shown in the following that a general theory can be developed, of which the vacuum-tube amplifier, the large-signal detector, the frequency converter and other devices are applications. A double-input superheterodyne mixer with straight input characteristics is discussed, and it is shown how the applied general theory predicts the necessity for non-linearity. The general theory is then used for the development of a substituting "model," from which important information on the behavior of a superheterodyne converter can be secured.

INTRODUCTION

THE mathematical treatment of non-linear devices may be simplified, if the current-voltage characteristic for the non-linear device can be represented by a series development containing terms no higher than the second degree. Thus small-signal detection theory appears in a simple form, if the detector characteristic is approximated to a parabola. Non-linear devices in general, however, produce outputs that can be accurately described only by a large number of terms or by means of unconventional mathematical functions. Still, in many cases, *the desired signal output is a linear function of the signal input.* This fact invites the replacement of the entire non-linear device by a linear "model," for which static parameters of one form or another are determined experimentally. Investigations of the non-linear device are then performed as investigations of the linear device; the only tool required being the simple mathematics for such a device.

Three variables and three parameters are generally required for the study of substituting "models": the input voltage (in case of ultra-high frequency devices the input power), the output voltage, and the output current. As a foundation for all types of "models," a general theory has here been developed in which, with reference to Fig. 1, the output current is chosen as the independent vertical variable, and the voltages as dependent horizontal variables. The "model" will then have the form of one or more current-voltage diagrams as is described in the following. The general theory is referred to as "the theory of related linear functions," as it is a straightforward application of plan-analytic geometry to the straight portions of the characteristics in the particular diagrams. The theory gives working formulae, from which the behavior of large-signal detectors, superheterodyne converters, etc., can be estimated and calculated. To demonstrate the principle, a linear device has been chosen as the first application to the theory and non-linear devices as second and third applications.

1. THE GENERAL THEORY

Assume a number of non-linear functions, such as

$$\iota = f'(\eta) \quad \text{for } \theta, \kappa, \cdots \text{ constant}, \tag{1}$$
$$\iota = f''(\theta) \quad \text{for } \eta, \kappa, \cdots \text{ constant}, \tag{2}$$
$$\iota = f'''(\kappa) \quad \text{for } \eta, \theta, \cdots \text{ constant}, \tag{3}$$
$$\cdots\cdots\cdots\cdots$$

The following discussion is general and includes any number of continuous functions, but for simplicity the treatment has been limited to the three functions given above.

Certain additional relations between the given quantities are introduced later, for example the inverse form of Eq. (3) $\kappa = f^{IV}(\iota)$, and the function $\kappa = f^{V}(\eta)$, for ι and θ constant.

The functions in Eqs. (1) to (3) are plotted in Fig. 1. If a point of operation Q_η is selected in diagram *a*, a horizontal line will locate similar points of operation Q_θ and Q_κ in diagrams *b* and *c*, all points representing one and the same ι-value ι_0. The slopes of the various characteristics in the Q-points are denoted k', k'', and k'''. Assuming linear regions around the Q points and small deviations $\Delta\eta$, $\Delta\theta$, and $\Delta\kappa$ within these regions,

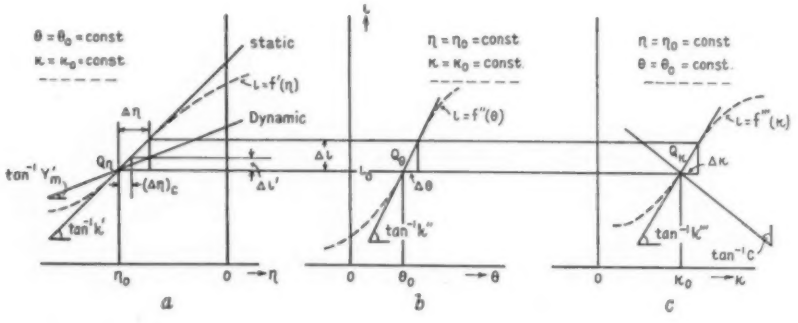

FIG. 1. Relations between three functions with common vertical variable.

the graph yields

$$\Delta\iota = k'\Delta\eta \quad \text{for } \theta, \kappa \text{ constant}, \tag{4}$$

$$\Delta\iota = k''\Delta\theta \quad \text{for } \eta, \kappa \text{ constant}, \tag{5}$$

$$\Delta\iota = k'''\Delta\kappa \quad \text{for } \eta, \theta \text{ constant}. \tag{6}$$

These are the related linear functions to be discussed. Obviously when κ is constant a change $\Delta\iota$ in the vertical variable ι can be produced either by a certain deviation $\Delta\eta$ in the horizontal variable η or by a certain deviation $\Delta\theta$ in the horizontal variable θ. If $\Delta\eta$ and $\Delta\theta$ take place in opposite directions, the vertical variable will remain constant. This fact makes possible the following definitions:

$$\kappa = \text{const.}, \quad k'\Delta\eta = -k''\Delta\theta, \quad -\frac{\Delta\theta}{\Delta\eta} = \frac{k'}{k''} = u'_{,,}. \tag{7}$$

Similarly for

$$\theta = \text{const.}, \quad k'\Delta\eta = -k'''\Delta\kappa, \quad -\frac{\Delta\kappa}{\Delta\eta} = \frac{k'}{k'''} = u'_{,,,}, \tag{8}$$

$$\eta = \text{const.}, \quad k''\Delta\theta = -k'''\Delta\kappa, \quad -\frac{\Delta\kappa}{\Delta\theta} = \frac{k''}{k'''} = u''_{,,,}. \tag{9}$$

To fulfill the requirement of linear operation around the Q points, it may be necessary to let the regions shrink to mathematical points. To define the slopes, Eqs. (4), (5), and (6) are written

$$k' = \left.\frac{d\iota}{d\eta}\right|_{\theta,\kappa}, \tag{10}$$

$$k'' = \left.\frac{d\iota}{d\theta}\right|_{\kappa,\eta}, \tag{11}$$

$$k''' = \left.\frac{d\iota}{d\kappa}\right|_{\eta,\theta}. \tag{12}$$

Similarly

$$u'_{,,} = \frac{k'}{k''} = -\left.\frac{d\theta}{d\eta}\right|_{\iota,\kappa}, \tag{13}$$

$$u'_{,,,} = \frac{k'}{k'''} = -\left.\frac{d\kappa}{d\eta}\right|_{\iota,\theta}, \tag{14}$$

$$u''_{,,,} = \frac{k''}{k'''} = -\left.\frac{d\kappa}{d\theta}\right|_{\iota,\eta}. \tag{15}$$

Equation (15) (analogous to (9)) can be obtained from Eqs. (13) and (14) (analogous to (7) and (8)) only if proper attention is given to the directions, in which the various deviations are counted.

It is important to realize that the defined coefficients are constants only if the characteristic curves in each diagram are straight, parallel, and equidistant within the region of operation. (Each diagram in Fig. 1 has a doubly infinite family of curves, each curve representing given parameter values. Only one curve or characteristic is shown in each diagram.)

If the principle of superposition holds, i.e., if the $\Delta\iota$ variations in Eqs. (4) to (6) remain the same even if the variations $\Delta\eta$, $\Delta\theta$, and $\Delta\kappa$ occur simultaneously, then the total $\Delta\iota$ variation $\Delta\iota_t$ is the sum of the contributions from the horizontal variables, so that

$$\Delta\iota_t = k'\Delta\eta + k''\Delta\theta + k'''\Delta\kappa + \cdots. \tag{16}$$

The horizontal variables may be any time functions arbitrarily related to each other. If they are assumed to be sinusoidal variations of the same frequency and of amplitudes* $|\Delta\eta_{\max}|$, $|\Delta\theta_{\max}|$, $|\Delta\kappa_{\max}|$, $\cdots$, related to a common reference quantity via phase angles α', α'', α''', $\cdots$, the instantaneous value of the vertical variable may be written

$$\begin{aligned}|\Delta\iota_{t\max}|\cos(\omega t+\gamma) = {} & k'|\Delta\eta_{\max}|\cos(\omega t+\alpha') \\ & + k''|\Delta\theta_{\max}|\cos(\omega t+\alpha'') \\ & + k'''|\Delta\kappa_{\max}|\cos(\omega t+\alpha''') + \cdots.\end{aligned} \tag{17}$$

This equation may equally well be expressed with sine functions. Multiplied by the operator j and added, the latter equation provides part of an equation system with orthogonal projections for fictitious, rotating complex quantities, so that in symbolic notation

$$\begin{aligned}|\Delta\iota_{t\max}|\exp j\gamma = {} & k'|\Delta\eta_{\max}|\exp j\alpha' \\ & + k''|\Delta\theta_{\max}|\exp j\alpha'' \\ & + k'''|\Delta\kappa_{\max}|\exp j\alpha''' + \cdots,\end{aligned} \tag{18}$$

or with capital letters for ι, η, θ, and κ used as symbols,

$$\mathrm{I}_{\max} = k'\mathrm{H}_{\max} + k''\Theta_{\max} + k'''\mathrm{K}_{\max} + \cdots. \tag{19}$$

This equation shows how the final variation in the vertical variable is built up in phase and

* In the figures amplitude is indicated by an eyebrow over the symbol rather than by the subscript max.

amplitude from the individual, primary, horizontal variations. If certain ties exist between the horizontal variables, so that H_{max} is the only truly independent variable, then I_{max} is fully described by diagram 1a, provided the static characteristic of slope k' is replaced by a so-called dynamic characteristic of slope Y_m'. It is sometimes practical to refer the entire study of the functions to one diagram only, and it will be shown how Eq. (19) may be written when referred to one diagram only. With the aid of Eqs. (7) and (8)

$$I_{max}=k'(H_{max}+\Theta_{max}/u_{,,}'+K_{max}/u_{,,,}'+\cdots)$$
$$=k'H_{c\,max}, \quad (20)$$

where

$$H_{c\,max}=H_{max}+\Theta_{max}/u_{,,}'+K_{max}/u_{,,,}'+\cdots. \quad (21)$$

The complex quantity $H_{c\,max}$, introduced as a notation for the parenthesis in Eq. (20), is called the $\iota\eta$ *control variable*, as this quantity alone controls the vertical variable ($\iota\theta$ and $\iota\kappa$ control variables could as well be developed). For the following discussion assume for simplicity that θ is kept constant and that K_{max} is a known function of I_{max}, for example, with C as a complex proportionality constant

$$K_{max}=-CI_{max} \quad (22)$$

(any sign may be assumed). Equation (19) then yields

$$I_{max}(1+k'''C)=k'H_{max},$$

so that

$$I_{max}/H_{max}=Y_m'=k'/(1+k'''C). \quad (23)$$

The complex ratio $I_{max}/H_{max}=Y_m'$ describes the dynamic characteristic, which in diagram 1a takes the place of the static characteristic k'. For the special case of C real, Y_m' degenerates from its general shape of an ellipse to a straight line, and is shown as a straight line in Fig. 1a.

With $u_{,,,}'$ from Eq. (8) substituted in Eq. (23), this latter equation will become of great importance for the applications that follow. Equation (23) may now be written

$$I_{max}=Y_m'H_{max}=u_{,,,}'H_{max}/(1/k'''+C). \quad (24)$$

A study of diagram 1a reveals that I_{max} may be obtained either from H_{max}, by using the true dynamic slope Y_m' or from $H_{c\,max}$, by using the original, static slope k'. This is evident from a comparison of Eqs. (20) and (24).

A similar discussion, applied to the quantities $\eta, \theta, \kappa, \cdots$ assuming a vertical deviation $\Delta\iota=\iota_0$, for the horizontal deviations $\eta_0, \theta_0, \kappa_0, \cdots$, yields

$$\iota_0=k'\eta_0+k''\theta_0+k'''\kappa_0+\cdots. \quad (25)$$

As before

$$\iota_0=k'(\eta_0+\theta_0/u_{,,}'+\kappa/u_{,,,}'+\cdots)=k'\eta_{0c}, \quad (26)$$

where

$$\eta_{0c}=\eta_0+\theta_0/u_{,,}'+\kappa/u_{,,,}'+\cdots. \quad (27)$$

η_{0c} is here the η_{0c} *control variable*. Even if Eqs. (25) and (27) are not fulfilled in practice, they give information as to the contribution of the various terms in creating a *positive* η_{0c}-value: a necessity in many applications, such as the following.

FIRST APPLICATION: THE LINEAR AMPLIFIER

The previously developed theory will now be applied to an amplifying tube such as the triode or pentode shown in Fig. 2a. The discussion will

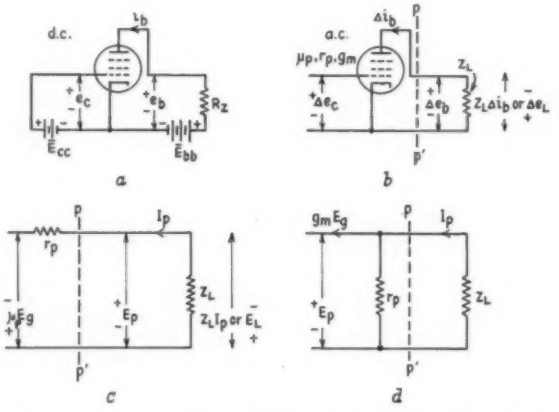

FIG. 2. Vacuum tube amplifier with equivalent circuits.

be limited to the case of one variational grid voltage and one variational electrode current—the plate current. It is assumed that the contributions in plate current from the various electrodes are in agreement with the principle of superposition. Therefore, in case of a pentode, the second and third grid are connected to cathode, directly or via capacitors.

The vertical variable ι, in the general theory represents the direct plate current i_b and the

horizontal variables η and κ may be chosen to represent the grid voltage e_c and plate voltage e_b. Figure 1a then illustrates the $i_b e_c$ diagram and Fig. 1c the $i_b e_b$ diagram. In accordance with Eqs. (10), (12), and (14) the tube coefficients are defined as

$$g_m = \left.\frac{di_b}{de_c}\right|_{e_b} = \text{transconductance}, \tag{10.1}$$

$$g_p = \left.\frac{di_b}{de_b}\right|_{e_c} = \text{plate conductance}, \tag{12.1}$$

$$\frac{1}{g_p} = r_p = \text{plate resistance}, \tag{28}$$

$$\mu_p = \frac{g_m}{g_p} = -\left.\frac{de_b}{de_c}\right|_{i_b} = \text{amplification factor}. \tag{14.1}$$

Relations between the variations Δi_b, Δe_c, and Δe_b are described by Eq. (16) with reference to Fig. 2b. For complex variables Eq. (19) yields the relation

$$I_{p\max} = g_m E_{g\max} + g_p E_{p\max}, \tag{19.1}$$

or with complex r.m.s. values

$$I_p = g_m E_g + g_p E_p. \tag{29}$$

This symbolic equation is probably the most important equation applying to the vacuum tube as far as circuit calculations are concerned. It is in the following referred to as the "tube equation."

If the plate voltage E_p is produced as the drop across the complex tube load Z_L, then

$$E_p = -Z_L I_p, \tag{22.1}$$

so that

$$I_p/E_g = Y_m = g_m/(1+g_p Z_L). \tag{23.1}$$

In case of a resistive load the path i_p *vs.* e_g degenerates from an ellipse to the straight line shown in Fig. 1a, i.e., from dynamic transadmittance Y_m to dynamic transconductance g_m.

In form of Eq. (24) the tube equation yields the well-known "Equivalent-Plate-Circuit equation" or "EPC equation"

$$I_p = \mu_p E_g/(r_p+Z_L), \tag{24.1}$$

where r_p, the tube resistance, replaces the inverse plate conductance g_p. In the practical application of this equation it is important to realize that the mathematical treatment is based upon the assumption of linear regions of operations around the Q points Q_η and Q_κ. If the diagrams 1a and 1c are combined, so that the characteristics form a space-model, this assumption means a flat surface of operation on this space model (constant tube coefficients).

The formulation of Ohm's law given in Eq. (24.1) may be represented as the equivalent circuit shown in Fig. 2c. As Eq. (24.1) is entirely positive, the e.m.f. $\mu_p E_g$ must aid the plate current, hence the given direction of the $\pm$ signs. The complex voltage gain A is easily obtained from Eq. (24.1) or from the equivalent circuit,

$$A = E_p/E_g = -\mu_p Z_L/(r_p+Z_L). \tag{30}$$

The equivalent circuit in Fig. 2c and its associated formulae refer to the series form of the EPC equation. The parallel form may be obtained if Eq. (30) is multiplied by E_g and g_m substituted for μ_p/r_p [Eq. (14.1)]. Then

$$E_p = -r_p Z_L/(r_p+Z_L) \times g_m E_g. \tag{31}$$

From this the equivalent circuit in Fig. 2d can be read off. Naturally $-E_L$ can be substituted for E_p in all the above formulae, if such is desirable.

The applications of formulae (20) and (21) are of interest as well. If all variational voltages are lumped to the signal grid, the variational electron current (here assumed equal to the variational plate current) is controlled entirely by the grid control voltage

$$(E_g)_c = E_g + E_p/\mu_p. \tag{21.1}$$

As is evident from Fig. 1a the plate current I_p may be obtained either from $(E_g)_c$ operating upon the static characteristic, or from E_g operating upon the true, dynamic characteristic. Equation (23) gives the relation between the slopes as

$$Y_m/g_m = 1/(1+Z_L/r_p). \tag{23.2}$$

Obviously the two characteristics coincide when $Z_L = 0$.

The "d.c. equation" (25) gives some information on the required values of applied potentials in tubes with many electrodes. For a pentode with the screen-grid voltage e_{sc},

$$(e_c)_c = e_c + e_{sc}/\bar{\mu}_{sc} + e_b/\bar{\mu}_p. \tag{27.1}$$

The $\bar{\mu}$ coefficients are here assumed evaluated to fit the equation and are identical with corresponding a.c. coefficients only for the never approached case of linear regions extended to zero plate current value. The control voltage $(e_c)_c$ must come out positive to produce an electron current. The grid voltage e_c is usually negative, and the last term negligible due to the high value of $\bar{\mu}_p$. The second term must therefore be made large enough to secure a sufficiently large value of $(e_c)_c$.

SECOND APPLICATION: THE LARGE-SIGNAL DETECTOR

The large-signal detector shown in Fig. 3

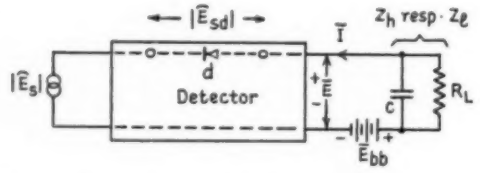

FIG. 3. Circuit for empirical determination of the detector coefficients.

operates with an input voltage

$$e_s = |E_{s\max}| \cos At, \tag{32}$$

$|E_{s\max}|$ being of the order of one volt or more. The load consists of a resistor R_L in parallel with a capacitor C, the d.c. value of the load being denoted R_Z, the a.c. impedance (in general) Z_L, the low frequency impedance Z_l, and the high frequency impedance Z_h. The applied voltage $|E_{s\max}|$ will develop a high frequency component across the load, which component will be considered negligible here, and a component $|E_{sd\max}| \doteq |E_{s\max}|$ across the non-linear element d. The conventional large-signal detector utilizes a diode as a non-linear element, but the treatment applies, within certain limits, to a contact-rectifier as well (for example, a silicon-crystal-tungsten-wire combination of the type used in ultra-high frequency work).

Without input wave the direct voltage across the load is either zero or very small. With input wave the device, operating in "Class C," furnishes the load with current pulses, which produce a steady direct voltage $\bar{E}$ across the load and a steady direct current $\bar{I}$ through the load. If curves $\bar{I}=f(|E_{s\max}|, \bar{E})$ where plotted, the diagrams obtained should be duplicates to any two diagrams in Fig. 1, for example 1a and 1c. Thus

$$\bar{I}=f'(|E_{s\max}|) \quad \text{for} \quad \bar{E}=\text{constant}, \tag{1.1}$$

$$\bar{I}=f'''(\bar{E}) \quad \text{for} \quad |E_{s\max}|=\text{constant}. \tag{3.1}$$

Figure 1c may now be replaced by Fig. 4a, as $\iota_0=\bar{I}$, $\eta_0=|E_{s\max}|$, and $\kappa_0=\bar{E}$. The characteristic curves in Fig. 4a are obtained from measurements. $|E_{s\max}|$ is given a certain value $|E'_{s\max}|$, and R_L, or the bias voltage $\bar{E}_{bb}$, or both, varied. The current $\bar{I}$ is read on a milliammeter for each setting, and the voltage $\bar{E}$ calculated (or vice versa), so that points a', a'', a''', $\cdots$ are determined in the diagram. The line joining these points is the desired characteristic $|E'_{s\max}|$. The procedure is then repeated for a number of $|E_{s\max}|$ values, until a family of curves is obtained. These curves can be used for any frequency for which the device behaves the same way as for the measurement frequency. A diagram obtained at 60 cycles cannot generally be used at ultra-high frequencies; $|E_s|$ can be corrected to $|E_{sd\max}|$, but the correction is difficult to make because of the varying impedance of the non-linear element (class C operation).

If during operation the voltage $\bar{E}_{bb}$ is gradually decreased, a point will be reached at which $\bar{E}=0$. If $\bar{E}_{bb}$ is decreased still further and made equal to zero, $\bar{E}$ will obtain a negative numerical value. The entire family of curves is now moved over on the left side of the vertical axis, so that the diagram will be of the form shown in Fig. 4. This is the conventional *rectification diagram* for a large-signal detector. The load

$$R_Z=R_L=-\bar{E}/\bar{I} \tag{33}$$

may in this diagram be represented by the load-line R_L, fixing a Q point Q at the intersection with the characteristic for the actually applied input voltage. (A load-line for $R_L'<R_L$ is as well shown in the diagram.) The Q point satisfies the steady-state conditions of the direct current $\bar{I}$ and the direct voltage $\bar{E}$.

In accordance with Eqs. (10), (11), and (14) the following definitions can be formulated

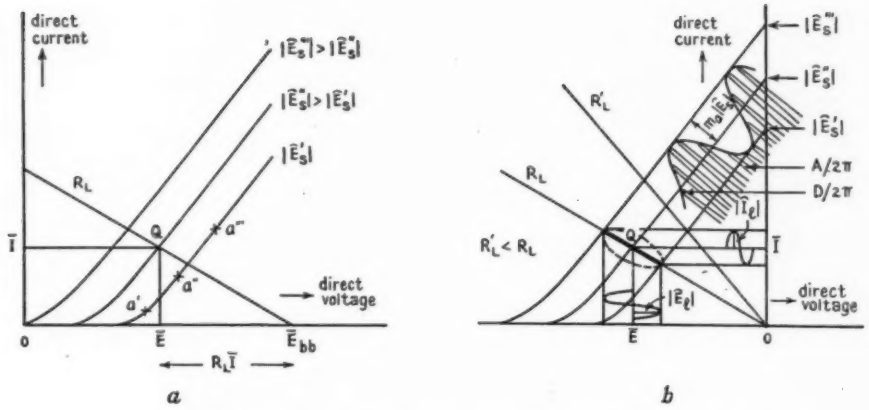

FIG. 4. Detection rectification diagrams, obtained from the circuit in Fig. 3.

$$g_d = \left.\frac{d\bar{I}}{d|E_{s\max}|}\right|_{\bar{E}}$$

$$= \text{detection transconductance}, \qquad (10.2)$$

$$\frac{1}{r_d} = \left.\frac{d\bar{I}}{d\bar{E}}\right|_{|E_{s\max}|}$$

$$= \text{detection conductance}, \qquad (11.1)$$

$$r_d = \text{detection resistance}, \qquad (34)$$

$$\mu_d = g_d r_d = -\left.\frac{d\bar{E}}{d|E_{s\max}|}\right|_{\bar{I}}$$

$$= \text{detection amplification factor}. \qquad (14.2)$$

These detection coefficients must be known for the following calculation and they are either measured or obtained from the rectification diagram. It is generally sufficient to obtain the coefficients from the diagram, where $1/r_d$ is the slope of the applied voltage characteristic at the Q point, and g_d is found by scaling off proper increments. The amplification factor may be obtained likewise, or from formulae (14.2) or both ways (for checking). μ_d can reach a maximum value of 1, obtained when $\bar{E} = |E_{s\max}|$.

The above discussion applies to the device used as a vacuum-tube voltmeter. For detector operation a modulated input wave is applied, such as

$$e_{sd} \doteq e_s = |E_{s\max}|(1 + m_a \cos Dt) \cos At, \qquad (35)$$

where m_a is the degree of amplitude modulation. The Q point will now slide along the load-line R_L, the path of operation being indicated by a heavy line, Fig. 4b. The sliding movement is determined by the amplitude and frequency of the modulation. The Q point will trace out variations $I_{l\max}$ and $E_{l\max}$ in $\bar{I}$ and $\bar{E}$, which variations constitute the output signal from the detector (l indicating low frequency). The output voltage

$$e_l = |E_{l\max}| \cos Dt \qquad (36)$$

is sinusoidal if the characteristics in the region of operation are straight, parallel, and equidistant. As the characteristics are crowded together at the origin 0, and curved near the horizontal axis, a large applied signal and a not-too-large load resistance with respect to the detector resistance are general requirements for low distortion. (Curved characteristics are permissible, however, as long as the detector load is truly resistive.)

Equation (22) in the general theory reads in this application

$$E_{l\max} = -Z_l I_{l\max}. \qquad (22.2)$$

$H_{\max}$ in Eq. (24) is the complex variation around the parameter value η_0 in Fig. 1c, respectively $|E_{s\max}''|$ in Fig. 4b. This variation is determined by the modulation to $m_a|E_{s\max}|$ and will now be used as reference quantity for the complex signal current $I_{l\max}$, which with the substitution in Eq. (22.2) is given by Eq. (24)

$$I_{l\max} = \mu_d m_a |E_{s\max}|/(r_d + Z_l). \qquad (24.2)$$

This "Ohm's law" is read off as the equivalent circuit, series form, shown in Fig. 5a. Here

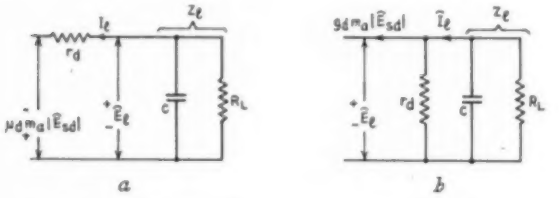

FIG. 5. Equivalent circuits for large-signal detector.

$\mu_d m_a|E_{s\max}|$ may be considered as the zero-phase-angle driving e.m.f., a sort of "equivalent voltage of detection." The circuit gives as output signal voltage

$$E_{l\max} = -Z_l/(r_d+Z_l)\times\mu_d m_a|E_{s\max}|, \quad (37)$$

which for $Z_l \gg r_d$, $\mu_d \doteq 1$ and $m_a = 1$ (100 percent modulation) simplifies to $|E_{l\max}| \doteq |E_{s\max}|$. This equality is never fulfilled in practical detector design. As an approximation $|E_{s\max}|$ indicates the largest AVC control voltage that can be obtained.

Equation (37), with $\mu_d = r_d g_d$, yields the relation

$$E_{l\max} = -r_d Z_l/(r_d+Z_l)\times g_d m_a|E_{s\max}|, \quad (38)$$

which is read off in form of the equivalent circuit, parallel form, shown in Fig. 5b. This circuit is not of the same general interest as the previous one, as the handy approximation $r_d \gg Z_L$ is seldom permissible. If an expression for the instantaneous output signal voltage is required, Eq. (36) may be written

$$e_l = -Z_l/(r_d+Z_l)\times\mu_d m_a|E_{s\max}|\cos Dt, \quad (39)$$

or

$$e_l = |-Z_l/(r_d+Z_l)| \times\mu_d m_a|E_{s\max}|\cos(Dt+\phi), \quad (40)$$

where ϕ is the phase angle for the impedance ratio. When ϕ deviates from π, the path of operation in Fig. 4b expands into an ellipse. As the capacitor C must be made large to make $Z_h \ll r_d$, the load cannot be made truly resistive, although zero load phase-angle, i.e., $Z_l \doteq R_Z$, is desirable from the point of view of low distortion. A discussion of distortion usually involves time-constant considerations, expecially if the signal diode is utilized to deliver the AVC control voltage as well. Even if the rectification diagram does not answer all questions regarding detector design, practice has shown that it is a great aid in practical detector design.

THIRD APPLICATION: THE FREQUENCY CONVERTER

Frequency converters are usually so non-linearly operated, that a straightforward mathematical treatment is rather difficult. A curved plate-current *vs.* signal-grid-voltage characteristic is a necessary requirement for proper functioning of so-called "sliding Q point converters" or "fixed path of operation converters" (example: a diode mixer plus oscillator), but is not a necessary requirement for so-called "shifting Q point converters" or "changing path of operation converters" (example: a pentagrid mixer plus oscillator).* It is possible, although not practical, to operate a shifting Q point converter in "Class A" with straight signal-grid as well as straight oscillator-grid characteristics. The general theory therefore applies to the signal input side of such a converter, but does not apply when the signal-grid and oscillator-grid act simultaneously.

A multielectrode type of tube with signal- and oscillator-grid separated by a screen-grid may be assumed to utilize electron coupling only (between signal-grid and the plate circuit and oscillator-grid and the plate circuit). In Fig. 1 ι may represent the plate current i_b, η the signal-grid voltage e_{cs}, θ the oscillator-grid voltage e_{c0}, and κ the plate voltage e_b. Figure 1a then shows the signal-grid characteristic and Fig. 1b the oscillator-grid characteristic, the variations in κ or e_b being assumed very small or equal to zero. Although the regions of operation in Fig. 1a and 1b may be straight lines, the total plate current variation is not described by Eqs. (16) or (19) because of the "gate effect." This effect causes the coefficients of one grid to vary with the voltage on the other grid, so that in addition to the two terms given by Eq. (19), a third so-called *frequency conversion term* will appear. The fact that the slope of the $\iota\eta$- or $i_b e_{cs}$ characteristic

* These technical terms have been adopted in one of the war training courses in electronics at Harvard University. This course will be available in book form at the end of 1943 or beginning of 1944. See as well H. Stockman, "Superheterodyne converter terminology," Electronics (Nov. 1943).

(i.e., the signal transconductance) now is a function of the oscillator voltage provides for over-all non-linearity. The action of the oscillator voltage upon the signal-grid transconductance may be visualized if diagrams a and b in Fig. 1 are combined in an orthogonal axes system, so that the regions of operation around the Q point form a characteristic surface. Figure 6 shows a volume element, extending from the Q point along the axes of voltage variations, and it is important to realize that the characteristic surface of this volume element is plane only in the case when there is no "gate effect." As this effect is present in arrangements with consecutive grids, Eq. (16)

$$\Delta\imath = k''\Delta\theta + k'\Delta\eta \tag{16.1}$$

must be replaced by

$$\Delta\imath = k''\Delta\theta + \left(k' + \frac{\partial k'}{\partial\theta}\Delta\theta\right)\Delta\eta, \tag{41}$$

or equally well by

$$\Delta\imath = k'\Delta\eta + \left(k'' + \frac{\partial k''}{\partial\eta}\Delta\eta\right)\Delta\theta. \tag{42}$$

For the same $\Delta\imath$ Eqs. (41) and (42) yield a relation, which when substituted into Eq. (42) gives this equation the form

$$\Delta\imath = k'\Delta\eta + k''\Delta\theta + \frac{\partial}{\partial\theta}\left(\frac{\partial\imath}{\partial\eta}\right)\Delta\eta\Delta\theta. \tag{43}$$

The coefficient in the third term—the frequency conversion term—determines the amount of frequency conversion obtained and illustrates the rate of change of the slope k' with an increment in the variable θ. This coefficient may be called the *non-linearity coefficient* e* and written

$$e = \frac{\partial^2\imath}{\partial\theta\partial\eta} = \frac{\partial^2\imath}{\partial\eta\partial\theta} = \frac{\partial k'}{\partial\theta} = \frac{\partial k''}{\partial\eta}. \tag{44}$$

Equation (42) gives with the above notations

$$\Delta\imath = (k' + e\Delta\theta)\Delta\eta + k''\Delta\theta, \tag{45}$$

where the parenthesis represents the dynamic slope Y_m' as a function of $\Delta\theta$. If the second term is considered negligible compared with the first one, then

$$\Delta\imath \doteq (k' + e\Delta\theta)\Delta\eta. \tag{46}$$

* This is the coefficient for the fifth term in a simple series expansion, hence the notation e.

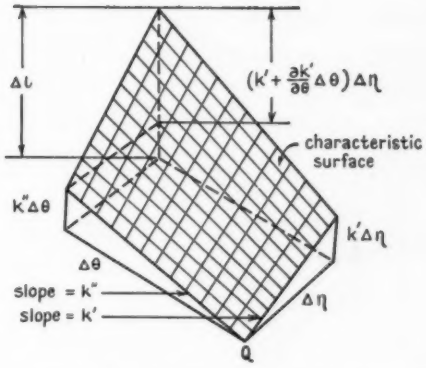

FIG. 6. Space model volume element to demonstrate the presence of a frequency conversion term.

This approximation illustrates the practical case of a mixer tube with $\Delta i_b = \Delta\imath$, $g_{ms} = k'$, $\Delta e_{c0} = \Delta\theta$, and $\Delta e_{cs} = \Delta\eta$, so that

$$\Delta i_b \doteq (g_{ms} + e\Delta e_{c0})\Delta e_{cs}, \tag{47}$$

or in terms of instantaneous values

$$i_p \doteq g_{ms}' e_{gs}, \tag{48}$$

where g_{ms}' represents the total signal-grid transconductance; the variational transconductance superimposed upon the steady-state transconductance. If the steady-state term is left without consideration and

$$e_{gs} = |E_{A\max}| \cos At, \quad g_{ms}' = e|E_{B\max}| \cos Bt,$$

the product term in Eq. (48) reads

$$i_p = e|E_{A\max}| \cos At\,|E_{B\max}| \cos Bt \tag{49}$$

$$= \tfrac{1}{2}e|E_{A\max}|\,|E_{B\max}| \cos (A-B)t + \tfrac{1}{2}e|E_{A\max}|\,|E_{B\max}| \cos (A+B)t. \tag{50}$$

The first or difference term is the i-f term in conventional superheterodyne designs.

This calculation is helpful for the understanding of frequency conversion, but does not aid particularly in practical design of converters with curved input characteristics and "Class C" operation. The more useful method of substituting a "model" for the converter will now be considered with reference to the results obtained above. The "large-signal detector" treatment of converters has been suggested by E. L. Chaffee, the static curves in a rectification diagram being used to predict the operation of the converter with a chosen load.

The block-diagram for a frequency converter is shown in Fig. 7. Here the comparatively large

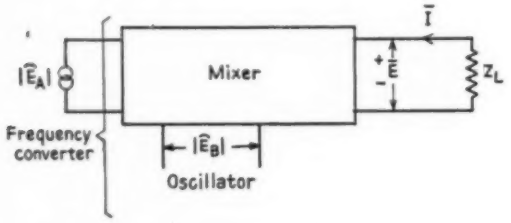

FIG. 7. Frequency converter circuit for empirical determination of converter coefficients.

oscillator voltage $|E_{B\max}|$ produces a periodic variation in the gain of a mixing device which has an input signal voltage $|E_{A\max}|$. Thus a product term $|E_{A\max}||E_{B\max}|$ is created in the output, yielding sum and difference frequencies. If the non-linear element exists in form of a diode, and the conditions $|E_{A\max}|=0$, $Z_L=R_L=100$ to 500 kilohms are fulfilled, then the circuit is a large-signal detector circuit of the type treated in the second application. The rectification diagram in Fig. 8 can be obtained if R_L is varied in steps and points a', a'', a''', $\cdots$ plotted. Any combination of an applied voltage $|E_{B\max}|$ and a load R_L determines a Q point with projections $\bar{I}$ and $\bar{E}$ representing the direct current and the direct voltage in the output.

A small input voltage E_A is now applied with the result that the heterodyning will produce sum and difference frequency currents, superimposed upon the steady value $\bar{I}$ (other direct and alternating currents being neglected). If the difference frequency were such a slow beat that d.c. instruments in the output could follow it, the variations of difference-frequency $C/2\pi$ indicated in Fig. 8 would result. These variations are determined by the positions of the points P and P', scaling off a part of the load line as path of operation. These points are here referred to as the "carrier point," respectively, the "image

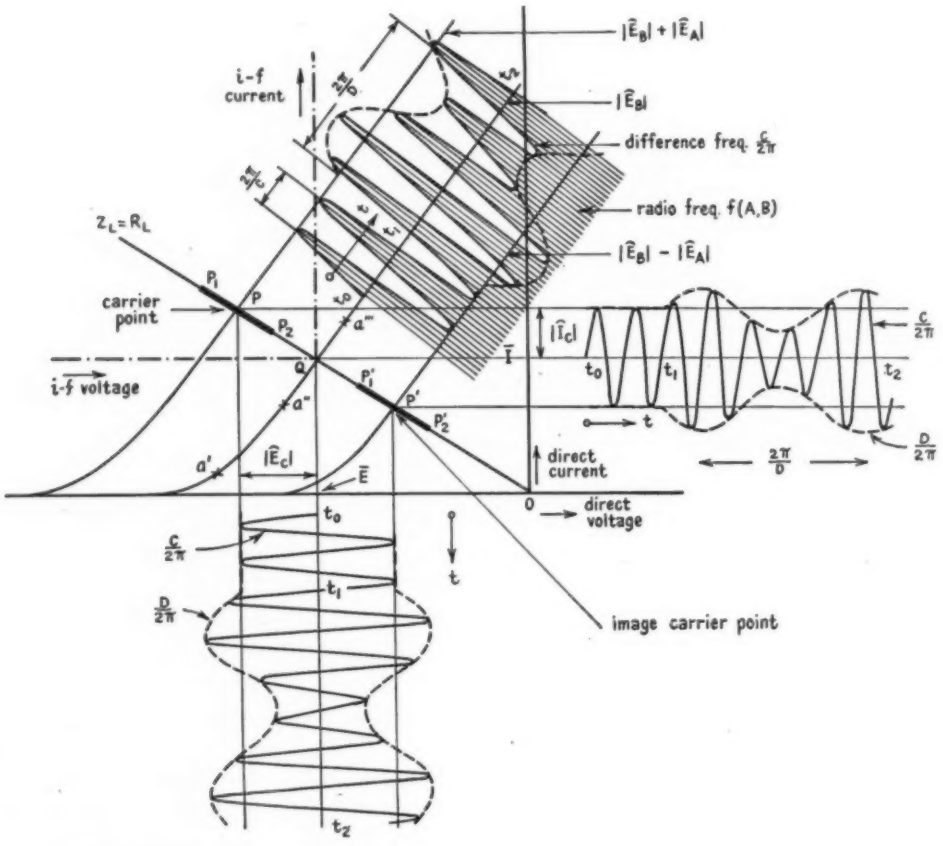

FIG. 8. Rectification diagram for frequency converter with modulated input wave.

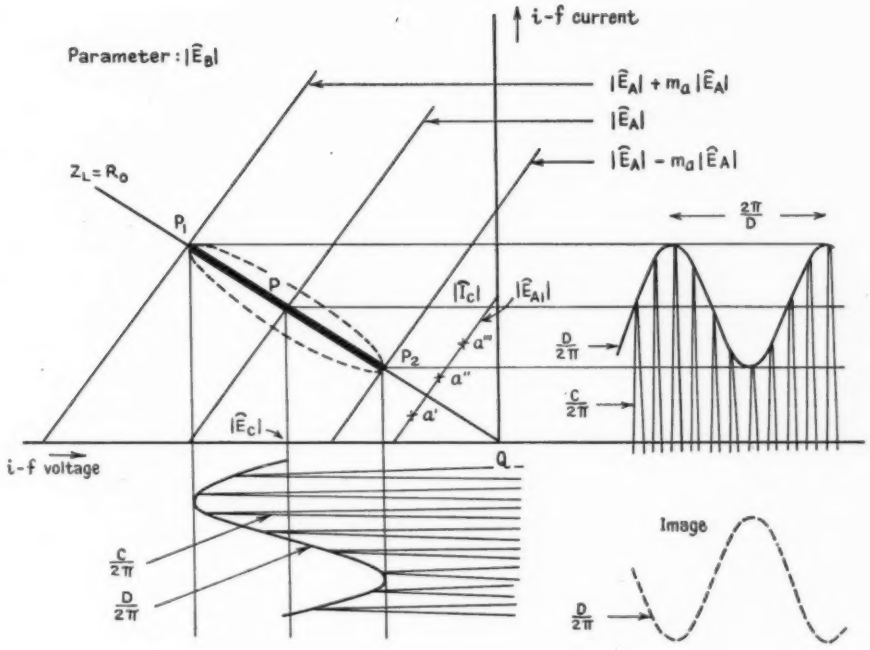

FIG. 9. Conversion diagram for frequency converter.

carrier point." The characteristics through these points are labelled $|E_{B\max}|+|E_{A\max}|$ and $|E_{B\max}|-|E_{A\max}|$. These expressions are true for a sliding Q point converter (such as a diode mixer plus oscillator), because the input to such a converter is a sum pattern of maximum and minimum values $|E_{B\max}|\pm|E_{A\max}|$. (The sum pattern is plotted in Fig. 8 with the characteristic $|E_{B\max}|$ as time axis.) The labelling of the characteristics does not apply to the physical converter when of shifting Q point type, but it gives the value of the oscillator voltage and signal voltage, to which the particular characteristic refers.

The current and voltage variations within the time interval t_0-t_1 represent the case of no modulation of the carrier voltage. If the incoming voltage $|E_{A\max}|$ is modulated by a sinusoidal tone of frequency $D/2\pi$, the points P and P' will slide back and forth along the load line and thus trace out the current and voltage variations shown within the intervals t_1-t_2. The envelope of the incoming wave therefore appears as the envelope of the outgoing wave, the contribution of the converter being merely to shift the frequency of the carrier.

As current and voltage variations are fully described by one of the two carrier points (assuming symmetry), the image carrier point P' will now be left out of consideration. If the axes system $|E_{c\max}|Q|I_{c\max}|$ is lifted out of the diagram in Fig. 8 and reproduced as the diagram in Fig. 9, a practical graphical representation results, which here will be referred to as the "conversion diagram." If in this diagram the load-line R_0 represents the input impedance to the i-f amplifier, when tuned to the converter output frequency, the other output components, including direct components, are of little influence (except in special cases). The conversion diagram can therefore be quickly obtained by means of i-f instruments attached to a variable i-f tuned impedance—the procedure being very similar to the one adopted for measurements on a large-signal detector. (Thus points a', a'', a''' $\cdots$ are obtained, which joined constitute one of the characteristics. Although, for simplicity, the characteristics in Fig. 9 are shown as straight lines, they may differ considerably in shape from large-signal detector characteristics.)

The path of operation due to tone modulation is indicated in Fig. 9 by a heavy line with the

end points P_1 and P_2. The characteristics through these points are labelled $|E_{A\max}|+m_a|E_{A\max}|$ and $|E_{A\max}|-m_a|E_{A\max}|$. Again these expressions are true when the diagram represents a sliding Q point converter, but symbolical when the diagram represents a shifting Q point converter.

The general theory is most practically applied if ι represents the i-f current $|I_{c\max}|$, η the applied voltage $|E_{A\max}|$, and κ the i-f voltage $|E_{c\max}|$. Thus the diagram in Fig. 9 corresponds to Fig. 1c. In the definitions given by the general theory the requirement of infinitesimal variations will now be replaced by a requirement of small a.c. amplitudes.

$$g_c=\frac{|I_{c\max}|}{|E_{A\max}|}\Bigg|_{|E_{c\max}|}^{\text{small ampl.}}$$

$$=\text{conversion transconductance;} \qquad (10.3)$$

$$\frac{1}{r_c}=\frac{|I_{c\max}|}{|E_{c\max}|}\Bigg|_{|E_{B\max}|}^{\text{small ampl.}}$$

$$=\text{conversion conductance,} \qquad (11.2)$$

$$r_c=\text{conversion resistance;} \qquad (51)$$

$$\mu_c=g_cr_c=-\frac{|E_{c\max}|}{|E_{A\max}|}\Bigg|_{|I_{c\max}|}^{\text{small ampl.}}$$

$$=\text{conversion amplification factor.} \qquad (14.3)$$

From Eq. (22) follows

$$E_{c\max}=-Z_LI_{c\max}=-R_0I_{c\max}, \qquad (22.3)$$

so that

$$|I_{c\max}|/|E_{A\max}|=|Y_m|=g_c/(1+R_0/r_c). \qquad (23.3)$$

The expression for the dynamic conversion transconductance Y_m is here of interest for absolute values, as $I_{c\max}$ and $E_{A\max}$ are of different frequencies.

The output current $I_{c\max}$ is given by Eq. (24) to

$$I_{c\max}=\mu_cm_a|E_{A\max}|/(r_c+Z_L). \qquad (24.3)$$

Then the plate impedance is useful as a complex quantity, as it determines the phase-angle of $I_{c\max}$ with $\mu_cm_a|E_{A\max}|$ as reference quantity. Equation (24.3) can be read off in form of the equivalent circuit in Fig. 10a, which produces an output voltage

$$E_{c\max}=-Z_L/(r_c+Z_L)\times\mu_cm_a|E_{A\max}|$$
$$=-r_cZ_L/(r_c+Z_L)\times g_cm_a|E_{A\max}|. \qquad (52)$$

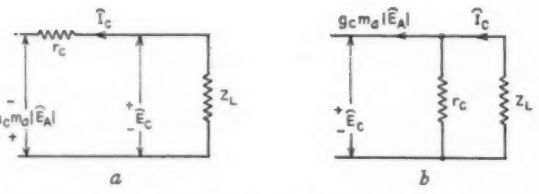

FIG. 10. Equivalent circuits for frequency converters.

If the conversion gain A_c is defined as the absolute ratio between the output i-f voltage and the input r-f voltage, then in agreement with Eq. (23.3)

$$|E_{c\max}|/|E_{A\max}|=|A_c|=|Y_m|R_0. \qquad (53)$$

This equation is general and applies to diode mixers as well as hexode and heptode mixers. In the latter case, however, the inequality $r_c\gg R_0$ makes possible the simplification

$$|A_c|\doteq g_cR_0. \qquad (54)$$

This equation is read off in form of the equivalent circuit in Fig. 10b with the shunting resistance r_c left out.

If the assumption $Z_L=R_0$ cannot be made, the path of operation will be an ellipse as is indicated in Fig. 9. When the equality $Z_L=R_0$ is fulfilled the ellipse degenerates to a straight line.

For a thorough investigation of the behavior of a certain frequency converter when $|E_{A\max}|$, $|E_{B\max}|$, R_0 supply voltages, etc., are changed, a number of converter diagrams such as the one shown in Fig. 9 are required. Contours with $|E_{B\max}|$ as independent variable can then be obtained from the individual conversion diagrams, and questions regarding optimum i-f amplifier input impedance and oscillator amplitude for maximum conversion gain, etc., answered. The variation of R_0 may, however, prove to be a problem in the experimental set-up, as tuning must be maintained during the measurements. It must first of all be decided if the conversion diagrams shall be obtained at very low frequency (method 1), at a medium frequency (method 2), or at the frequency of operation (method 3). Each method has advantages as well as disadvantages. A 60-cycle measurement ($A=2\pi60$, method 1) is scarcely possible as the difference frequency will be of the order of a few cycles. In addition the mixing

element may behave very differently at the much higher frequency of operation. Measurements at the frequency of operation (method 3) will eliminate this trouble but are rather delicate to carry out in case of mixers for centimeter waves. Especially in the case of an ultra-high frequency converter the method of obtaining the conversion diagram at a ten, hundred, or thousand times lower frequency than the frequency of operation (method 2) seems most attractive. It is then necessary to employ additional calculations and measurements to find the corrections, which will make the conversion diagram valid for the higher frequencies used in practical operation.

If the measurement frequency is just above the audio range and the converter is of broadcast or short wave type, for example, a triode-hexode, it is difficult to give the load impedance such a high impedance value that the bottom part of the conversion diagram will be covered. This difficulty may not exist for the low impedance mixers used in ultra-short wave converters. The variation in R_0 may be produced by L/C variation, by the introduction of series or parallel resistors or in case of a transformer by variation of the coupling.

CONCLUSION

It has been shown that design formulae for non-linear devices such as large-signal detectors and frequency converters can be obtained from a general theory for related linear functions. This treatment makes it unnecessary to use the vacuum tube theory (with its predetermined meanings of the tube coefficients) as a foundation for rectification and conversion diagram methods —a rather artificial foundation in case of crystal detectors and crystal converters. Naturally, if the total differential is written down for each case independently, a common theory is not necessary, but the similarities between the treatments are then less obvious.

The extension of the rectification diagram method to frequency converters seems not to have been covered previously in the literature. This extension provides a short cut around extensive and difficult calculations and changes the nature of experimental investigation to a standardized procedure. Some of the difficulties encountered in obtaining conversion diagrams are pointed out, and some hints given for the practical measurement work.

The contribution of this method is very similar to the contribution of the method of using $E_{g\max}E_{p\max}$ diagrams and contours in the power tube field. Diagrams are obtained for the tubes operating at a lower frequency, and it is possible to predict from these diagrams how the tubes will behave at a higher frequency and with a specified load.

ACKNOWLEDGMENT

Without the prior work by the Director for Cruft Laboratory, Professor E. L. Chaffee, on conversion diagrams obtained from a total differential, the writing of this paper would have been impossible. The author wishes to acknowledge his indebtedness to Professor Chaffee and to faculty members of Cruft Laboratory, who have contributed valuable suggestions.

APPENDIX 1: LIST OF PRINCIPAL SYMBOLS

Given as they appear in text. All deviations measured from average values.

I, ι	vertical variable (Iota)
H, η	horizontal variable (Eta)
Θ, θ	horizontal variable (Theta)
K, κ	horizontal variable (Kappa)
ι_0, η_0, etc.	steady values of above variables
k', u_{11}', etc.	real proportionality constants
C	complex proportionality constant
$d\iota$, etc.	infinitesimal variations in above quantities
$\Delta\iota$, etc.	finite variations in above quantities
$\Delta\iota$, I, etc.	complex r.m.s. value
$\Delta\iota_{\max}$, $I_{\max}$, etc.	complex amplitude value
$\lvert\Delta\iota\rvert$, $\lvert I\rvert$, etc.	absolute r.m.s. value
$\lvert\Delta\iota_{\max}\rvert$, $\lvert I_{\max}\rvert$, etc.	absolute amplitude value
ω	general notation for angular velocity
A, B, C, D	special notations for angular velocity
γ, ϕ, α', etc.	phase angles
Y_m	dynamic slope, dynamic transadmittance
$H_{c\max}$, etc.	complex $\iota\eta$ control variable
η_{0c}, etc.	direct $\iota\eta_0$ control variable
$\bar{E}_{cc}$	grid power-supply voltage
$\bar{E}_{bb}$	plate power-supply voltage
i_b	total instantaneous plate current
e_c	total instantaneous grid voltage
e_{sc}	total instantaneous screen-grid voltage
e_b	total instantaneous plate voltage

$(e_c)_c$	total instantaneous grid control voltage. (The above total quantities may represent d.c.)
di_b, etc.	infinitesimal variations in above quantities
Δi_b, etc.	finite variations in above quantities
I_p, E_g, etc.	complex tube-currents and voltages, r.m.s. values
g_m	transconductance, amplifier
$g_p = 1/r_p$	plate conductance, amplifier
μ_p	amplification factor, amplifier
A	complex amplification
R_Z	d.c. load impedance
Z_L	a.c. load impedance, $Z_L = R_L + jX_L$
Z_h	high frequency impedance of load
Z_l	low frequency impedance of load
r_d	detection resistance, large signal detector
g_d	detection transconductance, large signal detector
μ_d	detection amplification factor, large signal detector
m_a	degree of amplitude modulation
$\bar{I}$, $\bar{E}$	steady state direct current and voltage
$d\bar{I}$, $d\bar{E}$, etc.	infinitesimal variations in above quantities
$E_{s\,max}$, $E_{l\,max}$	complex amplitude voltages of absolute amplitude values $\lvert E_{s\,max}\rvert$, $\lvert E_{l\,max}\rvert$ and instantaneous values e_s, e_l. Corresponding complex r.m.s. voltages are E_s, E_l of absolute r.m.s. values $\lvert E_s\rvert$, $\lvert E_l\rvert$
$E_{sd\,max}$	component of $E_{s\,max}$ across device d
$I_{l\,max}$	complex amplitude signal current $E_{l\,max}/Z_l$
$E_{A\,max}$	complex amplitude signal voltage
$E_{B\,max}$	complex amplitude oscillator voltage
$E_{c\,max}$	complex amplitude voltage, i-f output
$I_{c\,max}$	complex amplitude current, i-f output
R_0	load impedance $Z_L = R_0 + jO$
r_c	conversion resistance, frequency converter
g_c	conversion transconductance, frequency converter
μ_c	conversion amplification factor, frequency converter
A_c	complex conversion amplification, frequency converter

A Compact High Resolving Power Electron Microscope

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(Received August 7, 1943)

In the early development of the electron microscope, the possibilities of high resolution microscopy by means of electrons were being explored and in the designing of the experimental instruments, greatest stress was placed on the critical operational components with little regard for the auxiliary apparatus and equipment. A number of instruments constructed during this stage of the development have been described in the literature.[1] Once it was shown that high resolution micrographs could be obtained by electron-optical methods and that the future of the electron microscope as a useful laboratory tool was assured, the methods of design underwent a gradual change which placed the stress on the reliability of performance, ease of operation, and simplicity of construction.

The next phase of the development was taken up mainly by industrial research organizations which were interested in making electron microscopes available for general use in scientific laboratories.[2]

An important landmark in this development was the conventional Type B microscope,[3] Fig. 1, developed by this laboratory in 1940 and now in use in many laboratories throughout the country. A major advance attained in this instrument was

[1] E. Ruska, Zeits. f. Physik **87**, 580–602 (1934); L. Marton, Bull. Acad. Belg., Cl. des. Sc. **21**, 606–617 (1935); A. Prebus and J. Hillier, Can. J. Research **A17**, 49–63 (1939).

[2] B. von Borries and E. Ruska, Wiss. Veroff. d. Siemenswerke **17**, 99–106 (1938); L. Marton, M. C. Banca, and J. F. Bender, RCA Rev. **5**, 232–243 (1940).

[3] J. Hillier and A. W. Vance, Proc. I. R. E. **29**, 167–176 (1941).

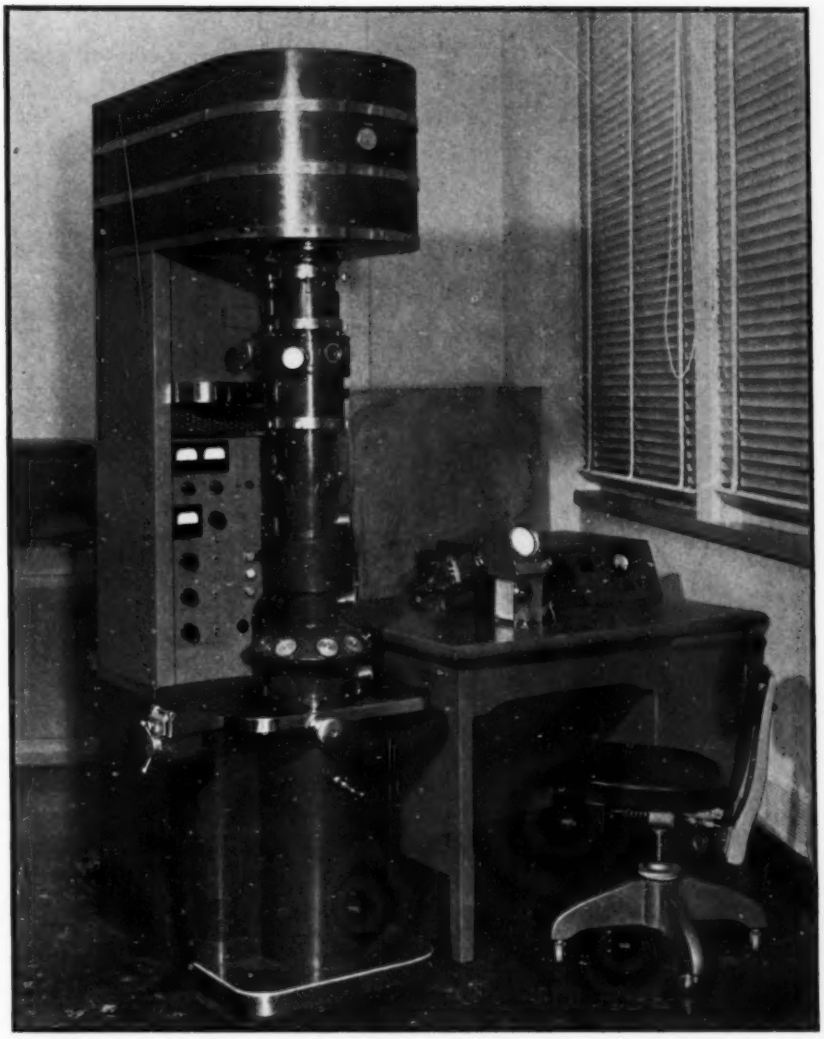

FIG. 1. A comparison photograph showing the conventional Type B microscope and an experimental model of the new small instrument.

the great reduction of the physical dimensions of the electrical power supplies, enabling them to be combined physically with the electron microscope proper. Some advance was also made by reducing the complexity of the instrument itself and the associated vacuum techniques. This microscope is able to produce electron micrographs showing separations as small as 20–30A[4] between small heavy particles. The consistency with which technically satisfactory micrographs may be obtained is also fairly high being between 60 and 70 percent.

[4] Recent unpublished results have shown that the resolving power of this instrument lies in the neighborhood of 25A.

As a result of our own experience and of numerous discussions which we have had with the users of electron microscopes, the development of the instrument has progressed into its third phase; namely, another type of instrument stressing simplicity and refinement. This further advancement in design has resulted in an instrument possessing a degree of performance that is consistent with simplicity of operation and servicing.

DISCUSSION OF DESIRED INSTRUMENT QUALITIES

In the laboratory design of a new electron microscope a number of primary factors are to be

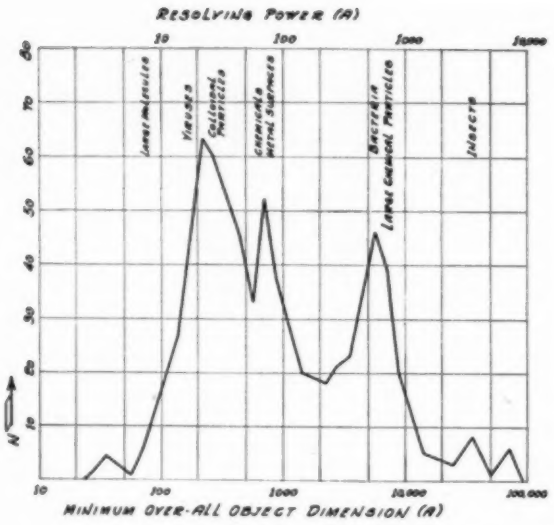

FIG. 2. A curve showing the relationship between the frequency of occurrence of applications of the electron microscope and the resolving power required.

considered; namely, the performance, simplicity of operation, and versatility. If the laboratory instrument is to pass easily into the subsequent stages of engineering and production, complexity of design features and applications in various laboratories must also be kept in mind.

The performance of an electron microscope can be divided into two parts: the electron optical performance; that is, the resolving power and penetration attainable with any given lens system and accelerating voltage; and the operational performance, which includes the complexity of manipulation and technical reliability of results.

In order to determine accurately an optimum value for the resolving power desired in this type of instrument, it would be necessary to know the requirements of the workers who would ultimately use it. While this is not possible, an estimate can be made by considering the problems to which the electron microscope has already been applied. Fortunately the past work of this laboratory is ideally suited for making the necessary survey. It has been our practice to demonstrate our electron microscopes to anyone who had a legitimate interest in them and in nearly every case a few micrographs were taken of materials in which these individuals were particularly interested. As a result of this, we have accumulated over the last three years close to 10,000 electron micrographs of every conceivable type of material. A survey was made of these electron micrographs and a number of micrographs selected which represent the type of specimens brought in by these scientific workers. The critical dimension on each of the 748 micrographs selected as representative from the total number available was measured and tabulated in the frequency diagram shown in Fig. 2. This diagram can be considered to give a fairly good picture of the range of application of the electron microscope at the present time. It can be seen to have three major peaks at approximately 200, 700, and 6000A. As marked on Fig. 2, these peaks correspond to viruses and colloidal particles in the first, chemicals and metal surfaces in the second, and bacteria and larger chemical particles in the third.

It will be noted from this diagram that the great bulk of electron-microscope work at the present time occupies the size-range between 100 and 1000A. The other peaks represent somewhat specialized and narrower applications. In order to use this diagram to determine the optimum resolving power of the new instrument, it was necessary to transform the scale of abcissae, which now represents the actual over-all dimensions of the objects of interest in the individual micrographs, to a scale which represents the resolving power actually necessary to image properly any given particle or structure.

In examining an object with the electron microscope we are interested primarily in obtaining information regarding one or more of three of its physical properties: size, shape, and structure. In considering measurements of size on electron micrographs, it becomes apparent that the major error in the measurements of small particles is the image diffusion caused by lack of resolving power. In fact, the percentage error to be expected can be expressed by the form $(100\Delta d)/d$, where d is the dimension in the object and Δd is the indeterminancy caused by the image imperfections. It is obvious that the resolving limit should be at least one order of magnitude below the dimension being determined if the error of measurement is not to exceed 10 percent. Borries and Kausche[5] have pointed out

[5] B. von Borries and G. A. Kausche, Kolloid Zeits. **90**, 132 (1940).

that the resolving power similarly limits our ability to determine shape in a micrograph. In fact, if we wish to distinguish the image of an eight-sided particle from the image of a sphere, he has shown that the resolving limit must be at least ten times lower than the physical dimension of the particle. An exactly similar situation exists with regard to determining structure.

Thus, by consideration of the important information which a micrograph can give, it is seen that it is absolutely necessary for the resolving limit of the imaging system to be at least an order of magnitude below the physical size of the structure or particles being examined. Hence, at the top of Fig. 2, a new scale of abcissae has been added which gives the resolving power necessary for each use of the electron microscope.

It is apparent from this new scale that the resolving limit of an electron microscope must lie below 20A if the instrument is to be applied in the size range for which there is a maximum number of problems to be studied. Actually, even the most elaborate existing instruments have not quite sufficient resolving power for adequate coverage of this size range. This is particularly true of organic materials where, for reasons of contrast, the resolving limit of the best present instruments may be only 50A. The examination of any good electron micrograph of virus particles will demonstrate this point. For instance in the case of tobacco mosaic viruses, Fig. 3, which are roughly 120A in the short diameter, it is obvious that in order to prove the existence or non-existence of internal structure, it would be necessary to have an instrument giving several times the resolution of the one with which this particular micrograph was made.

Consequently, in the development of a new microscope for the widest possible range of application our reasoning leads us to desire the best resolving power attainable at the present time.

Assuming a resolving power of 80 lines per millimeter for the photographic emulsion used, we find that a resolution of 20A requires an instrumental magnification of 6000× if all the details present in the image are to be recorded. This value of magnification has been deduced entirely from the relative resolving powers of a given electron optical system and a photographic emulsion. There are, however, other factors based on operational considerations which must be taken into account before the final decision is made as to the most useful magnification.

The Type B electron microscopes give a range of magnification between 1000× and 20,000× in a series of steps. During the operation of this instrument in the past three years, a number of interesting facts have become apparent: (1) The value of the lower end of this range of magnification is limited by the smallness of the field of view obtainable (roughly 10 microns in diameter). (2) Most of the workers favor a magnification somewhere between 6000× and 10,000× and keep the instrument at whatever value is chosen throughout most of its operation. (3) A high value of magnification—around 20,000×—has been found to be desirable in a number of exceptional cases. These usually involve photographs of extremely fine particles which cannot be distinguished easily on the final fluorescent screen at the lower magnifications. (4) Discussions with the users of the electron microscope have shown that a relatively low magnification of about 500× and a correspondingly large field of about 150 microns is also very desirable as it permits easy comparison between electron micrographs and ordinary photomicrographs of identical fields of view.[6] (5) Recently, metallurgists using the replica technique have found the magnification range 2000–2500 most useful.

FIG. 3. A highly enlarged electron micrograph of tobacco mosaic virus demonstrating the need for greater resolving powers. 82,500×.

[6] C. J. Burton, R. B. Barnes, and T. G. Rochow, Ind. Eng. Chem. **34**, 1429–1436 (1942).

After analyzing the whole situation with regard to the new microscope it was realized that most of the advances which could be made with regard to instrument size, field of view obtainable, and instrument complexity, depended on keeping the electronic magnification as low as possible. In view of the above observations it also became apparent that, while a continuous range of magnification is convenient, it is not absolutely essential to the optimum performance of the instrument. However, two values, one an order of magnitude less than the other, are desirable. The low magnification was set at between 500× and 600× because at this magnification a single opening of the standard 200-mesh screen is recorded in its entirety on a standard 2″×2″ lantern slide, while the high magnification was set at 6000× for the reasons given above.

The conditions that led to the determination of the electronic magnifications to be provided in the new instrument also indicated the optimum arrangement of the components as well as simplified operating techniques. For instance, the fact that a continuously variable range of magnification is not necessary, leads directly to the possibility of combining the projection and objective lenses into a unit excited by one coil winding and one magnetic circuit. Having fixed the magnification and specified that a single specimen position and a single value for the accelerating potential be used, it is obvious that the focal lengths of both the projection and objective lenses will be fixed. In actual operation, of course, it will be necessary to vary the focal length of the objective over a small range for focusing the instrument. Since the focal length of each lens can be adjusted in the design of the pole pieces themselves independently of the exciting magnetic flux, the use of a single exciting coil and magnetic circuit becomes an immediate practical possibility. This type of design is very attractive, for not only does it cut the mechanical construction down to a fraction of that in the system consisting of two independent lenses but it also completely eliminates one regulated current supply. It also increases the attainable precision of mechanical alignment of the two lenses, thus permitting the elimination of a mechanical adjustment. The only operational difference from the conventional arrangement is that the strength of the projection lens, and hence the magnification, varies slightly when the objective lens field strength is adjusted to obtain exact focus. However, the rate of variation of the magnification with change in field strength is only twice as great as it would be if the objective and projection-lens-fields were independent of each other. Low magnification is obtained by properly modifying the projection-lens pole pieces. Another attractive possibility which comes as a result of a fixed and relatively low electronic magnification is the complete elimination of the magnetic condenser lens and its associated power supply.

Up to the present time high resolution electron microscopes have been designed with a vertical optic axis; the electron gun is usually placed at the top and the viewing screen at the bottom. This arrangement is mainly the result of the weight of the components and the simplicity which it affords in the design of the alignment adjustments and the photographic chamber.

In this arrangement it is necessary to view the fluorescent screen from the same side as the incident beam making it difficult to use a satisfactory light-optical stage of magnification for focusing the instrument. In the new instrument, with its fixed magnification, the use of an additional light-optical stage of magnification is necessary. This led to the use of a transparent fluorescent screen viewed from the side opposite to the incident beam; a possibility which was finally contingent on the elimination of all adjustments between lenses, on the contemplated dimensions of the instrument, and on a number of other factors.

A change in design as radical as that indicated in the foregoing discussion must of necessity require a new technique of operation. It is, therefore, necessary to determine whether the required changes provide the desired increase of simplicity. In the operation of vertical type microscopes the practice has been to examine the field of view and focus the instrument visually with relatively high intensity. Thus, during focusing, the angular aperture of the illuminating beam is large and the depth of focus correspondingly small, while after focusing and prior to making the exposure the angular aperture of the illumination is usually

reduced by nearly an order of magnitude. Theoretically, this represents an ideal method of operation since the focusing is done with small depth of focus while the photographic record is made under conditions of large depth of focus. This enables the operator to compensate for the fact that his eye cannot distinguish the finest details of the electronic image and hence, he cannot focus the instrument with absolute accuracy. In practice a number of phenomena prevent the functioning of the instrument from conforming exactly with the theory. For instance, the change in intensity of the illumination changes the charged-up condition of the specimen and of any insulating films or particles which may be adhering to the walls along the path of the electron beam thus causing the final image to shift. The time required for the system to reach a new stage of equilibrium may extend through the exposure, resulting in a spoiled picture. The fact that the space-charge condition in the beam at the specimen also depends on intensity, gives rise to a change in focus of the entire system requiring the objective current to be set slightly below the value for visual focus if the best possible record is to be obtained.

These are, in general, second-order effects and enter into the results only when extreme demands are made on the resolving power of the instrument. Moreover, they can be completely eliminated by the method of series exposures. In the new instrument the relatively low magnification requires even greater accuracy of focusing, yet the restrictions on complexity made it appear impractical to incorporate the construction necessary for attaining series exposures. Therefore, an alternative method is necessary. In this case the intensity of illumination is maintained constant throughout the complete operation of focusing and photographic exposure. The final arrangement of the screen is such that with a 20× eye lens a bright image at about 100,000× can be observed visually. Under these conditions it is possible to focus the instrument with extreme accuracy even for the smallest observable particles and also to detect any image errors which may arise as a result of a defective source, a dirty lens system, or motion of the specimen.

The adjustments required constitute another factor which enters into the development of a new electron microscope. In earlier instruments some form of alignment adjustment has always been incorporated because it enables the operator to compensate for inaccuracies of machining and for the deflection effects of charged particles on the lens openings. The technique of alignment, while not difficult, requires a certain amount of time and practice and since conditions in any given instrument may change from time to time, a fairly good understanding of the principles involved is necessary if the instrument is to be put rapidly into its best condition for operation. As routine observation for control purposes is considered to be an important future use of electron microscopes, it has been apparent that alignment adjustments should be eliminated as much as possible. Actually, the small dimensions involved in the type of design contemplated, so reduced the sensitivity of the instrument to the disturbing effects that the necessity for alignment was automatically eliminated.

The use of a transmission-viewed screen provides two possibilities with regard to the photographic recording technique. Namely, outside photography of the fluorescent-screen image by means of a camera mounted externally to the instrument or direct internal photography. Both methods have been used in the past and both have advantages and disadvantages which must be evaluated before a choice can be made. Recent tests both in this laboratory and elsewhere[7] indicate that the resolution obtainable in an electronic image observed on a fluorescent screen is somewhat better than 100 lines per millimeter. In order to demonstrate a resolving power of 20A a magnification of only 5000× must be obtained on the fluorescent screen. Thus, assuming that no definition is lost in the lens system of an external camera it appears that the resolving powers of the two methods are comparable. The average exposure time for a high resolution micrograph using Eastman medium-lantern-slide emulsions and internal recording is about five seconds at a magnification of 6000×. This short exposure time has great advantage in the elimination of occasional external disturbing effects. In the

[7] C. H. Bachman and S. Ramo, J. App. Phys. **14**, 69–77 (1943).

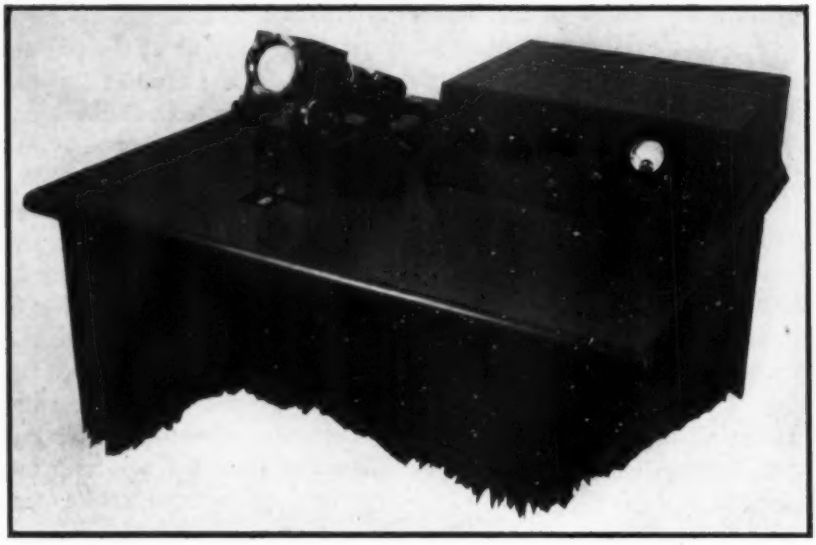

FIG. 4. A photograph of the experimental model of the small electron microscope.

literature[7] it has been indicated that the exposure time for external photography of the fluorescent screen image at 500× is about 2 minutes. Since the intensity of the fluorescent image varies inversely as the square of the electronic magnification, the corresponding exposure time for external photography of a high resolution image would be a number of hours. A still further increase of exposure results from the necessity of using a narrower angular aperture in the illuminating beam to obtain high resolution. For internal photography it is necessary to insert in the vacuum chamber a new photographic plate for each exposure or each group of exposures, while in the external method this time is completely eliminated. A survey of a number of present users of electron microscopes has shown that, for a large part of their work, only one plate of exposures is made of any given specimen. In these cases the photographic plate and specimen are changed simultaneously and the pumping time enters for either method.

Thus, it appears that the actual decision between the two methods depends on the ultimate resolution desired. At 20A external photography with the present screen materials is out of the question, while at 200A the decision is difficult to make because the longer exposure time for the external method plus increased effects of disturbances are more or less balanced by the longer time of manipulation required with the internal method. For still lower power work, which would be of a survey nature, the external method is probably advantageous.

In investigating the efficiency of fluorescent materials for use on electron-microscope screens a fairly extensive survey of available phosphors was made. A number were found which gave a three- to fourfold increase in the image intensity as compared to that of the willemite commonly used. Using these materials on the new small instrument has resulted in a fluorescent image intensity, at a magnification of 5000×, of the same order as that of a bright television picture. With a simple light shield the image can be viewed quite easily in a well-lighted room.

There were also a number of other factors which affected the complexity and versatility. These included the use of a 30-kilovolt accelerating potential, the use of a simple single-stage diffusion pump, and the substitution of a variable focusing aperture potential for the conventional condenser lens. Further reductions in complexity were obtained through normal engineering development particularly by simplification of the electrical power supply.

THE EXPERIMENTAL DESIGN OF A SMALL ELECTRON MICROSCOPE

Figure 4 is a photograph of the present experimental model of a small electron microscope. Figure 5 is a close-up of the microscope proper while Fig. 6 is a simplified cross section. It should be pointed out that the model described here has reached its present form as a result of innumerable modifications and, hence, it contains a number of elements of purely experimental character which have to be completely redesigned before the instrument can be produced commercially. As the purpose of our present instrument and of this paper is purely the demonstration of electron optical and design principles, the details which are described below are those of a laboratory model.

The electron microscope proper is built as a rigid unit and mounted at an angle of 20 degrees with respect to the horizontal with the electron gun at the lower end. The valve block and oil-diffusion pump are designed as an integral part of this unit. It is mounted, in the present model, on a standard small-size stenographic desk so that the final viewing screen is at the most comfortable viewing position for a seated operator. A large lens providing three times optical magnification can be placed in front of this screen so that the operator is effectively viewing a field of vision 9″ in diameter at a total magnification of 15,000 and at an intensity slightly greater than that observed on the screen itself. The remainder of the microscope equipment, including the me-

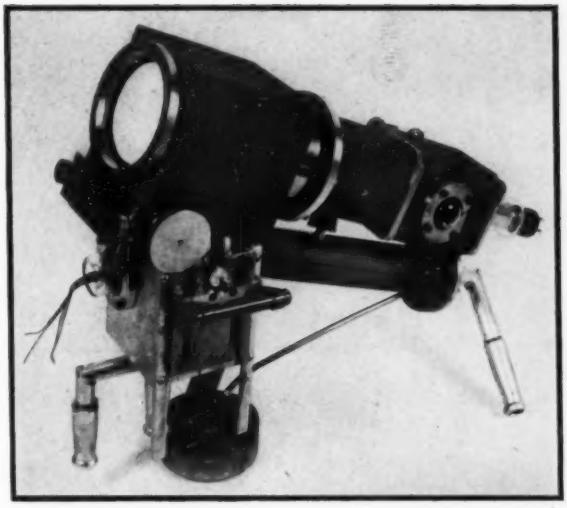

FIG. 5. A photograph of the microscope unit showing the valve block and diffusion pump.

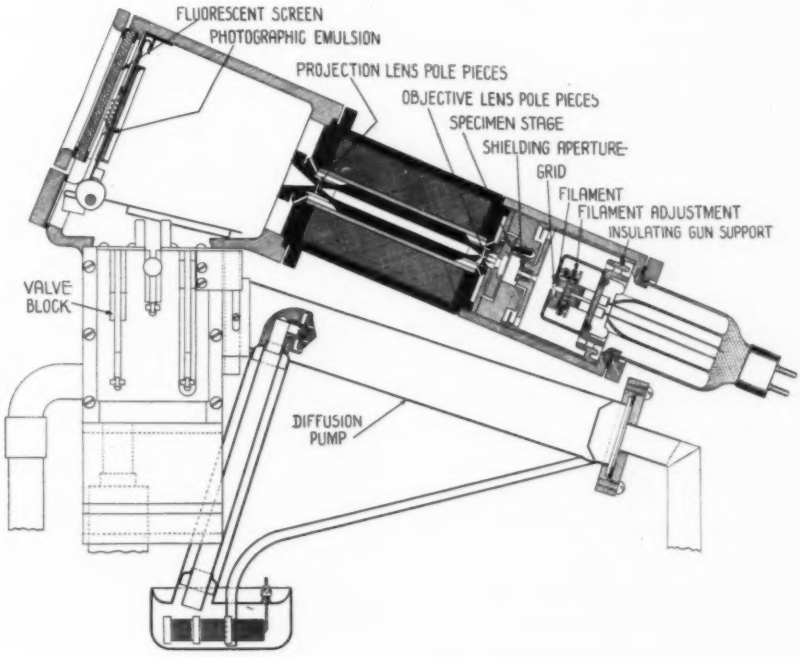

FIG. 6. A simplified cross section of the small electron microscope unit.

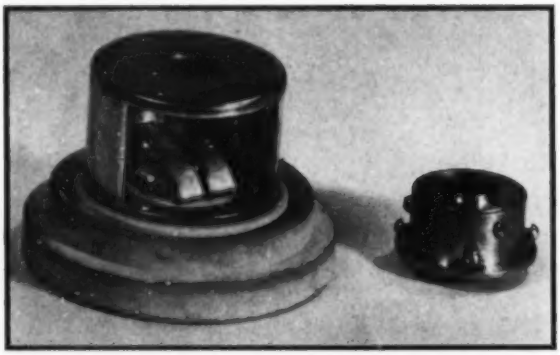

FIG. 7. A photograph of the cathode structure showing the filament capsule removed.

chanical vacuum pump, is mounted within the confines of the desk.

The electron gun consists of the usual hairpin filament heated by 60-cycle alternating current. The cathode structure consists of a small removable capsule which includes the focusing aperture and an adjustable filament support (Fig. 7). The filament is 0.004″ diameter tungsten wire which is threaded through the filament supports, properly formed over a removable jig and clamped into place. The whole filament assembly can be adjusted by means of four adjustment screws so that the filament tip is coaxial with the focusing aperture. When a new filament is inserted this adjustment is made visually and the capsule inserted in the microscope. After obtaining a vacuum the filament is heated to its normal operating temperature for about a minute to allow it to take up an unstrained position. It is then removed from the microscope and recentered. The results up to the present time have indicated that after this second adjustment the filament maintains its position throughout the rest of its life. This means of alignment was found to be sufficiently accurate for the making of photographic records. However, in the actual operation it was found that if the focusing aperture potential was adjusted so that beam crossover coincided with the specimen plane, the actual area of the specimen illuminated was usually slightly off the field of view of the objective. As the high image intensity which could be obtained by proper alignment of the gun at this adjustment was exceedingly attractive and since it did not seem possible to attain sufficiently precise prealignment, a slight amount of tilting adjustment has been incorporated in the electron gun. The axial position of the filament is that for which the cathode assembly produces a well-focused image of the emitting area at the plane of the specimen, the focusing aperture being maintained at the same potential as the filament.

The potential of the focusing aperture can be varied from 0 to 75 volts negative with respect to the filament. As this is sufficient to place the image of the filament produced by the system within a few millimeters of the cathode assembly, an ideal illuminating beam is obtained. In view of the fact that the angular aperture of this illumination is between 10^{-3} and 10^{-4} radian, maximum definition is obtained in the recorded image. When the focusing aperture potential is adjusted so that the image of the filament is focused on the specimen, very high intensity is obtained which is ideal for visual observation. Thus it can be seen that the adjustable focusing aperture potential performs the same function as is performed in the conventional microscope by the condenser lens, enabling the latter to be eliminated.

In the new instrument, however, the area of irradiation is about 1 millimeter in diameter as compared with 0.025 millimeter when a condenser is used. Since the intensity of irradiation is roughly the same in both cases the actual

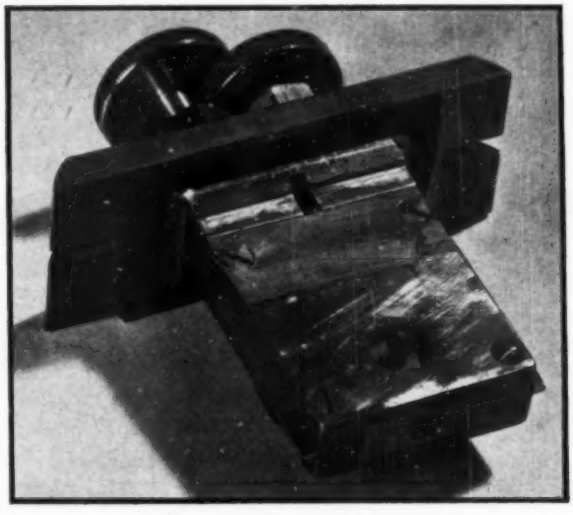

FIG. 8. The photograph of the specimen holder and movable specimen stage removed from the instrument.

electron energy which impinges on the specimen is much higher in the former. While this electron energy passes through the thin membrane of the specimen without appreciable loss, it is almost completely absorbed by the wires of the supporting screen. It was found that these wires were not capable of conducting the resulting heat into the body of the microscope with sufficient rapidity and hence they often raised the temperature sufficiently high in extreme cases to melt or distort the screen wires and always high enough to melt the supporting membrane at the point of contact, resulting in its rupture. This difficulty was finally eliminated by physically limiting the beam diameter by a small diaphragm placed immediately before the specimen.

SPECIMEN STAGE

The specimen-stage mechanism is of a design which has been found most satisfactory during the past two years (Fig. 8). It consists essentially of a movable plate mounted on ball-bearings in such a way that it can be moved in a plane normal to the axis of the instrument. The desired motion is transmitted to it through two adjusting screws which are threaded into the body of the specimen chamber and in the case of the present instrument simply have large knobs on the outside for manipulation. The vacuum is maintained by means of a packing gland type of seal. In the present instrument a circular area of the specimen 2.5 millimeters in diameter can be scanned. The return motion for the stage adjustments is provided by a single helical spring placed midway between the adjusting screws and working under tension. The specimens are mounted on standard one-eighth-inch disks of 200-mesh screen and are clamped on the end of a tubular projection of the movable stage by means of a small cap. In the present model the entire stage and adjusting screw mechanism is removed as a single unit from the side of the instrument each time the specimen is changed. This type of structure was used in order to facilitate the development of the mechanism. Except for the ease of experimentation which it provides, it has no advantages over the type of design in which the specimen stage and adjusting mechanism are an integral part of the microscope body. In further developments the latter type of construction will undoubtedly be used. The adjusting screws were tentatively designed so that one revolution of the external handle resulted in 0.8-millimeter displacement of the specimen. This rate of motion has been found highly satisfactory in our own laboratory particularly in that it allows the specimen to be

FIG. 9. A photograph of an experimental pole-piece unit.

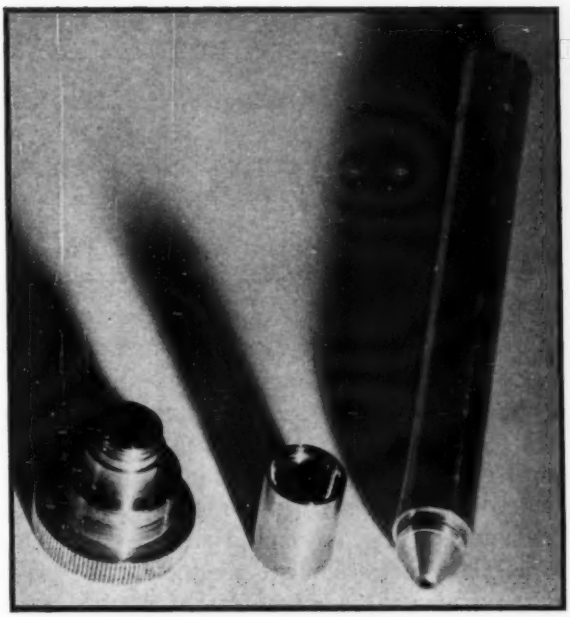

FIG. 10. A photograph of the same pole-piece unit with one end disassembled to show its construction.

scanned very rapidly and yet it is sufficiently fine to allow any point of the specimen to be brought easily to the center of the field at the highest magnification. This type of object chamber mechanism can be easily modified to enable stereo-micrographs to be taken.

LENS SYSTEM

The magnetic lens system used in the present model is a result of a long series of developments and, like other parts of the instrument, has some features which were incorporated to facilitate development but normally will not be used. The coil winding and external magnetic circuit is quite conventional except that in the present model it has been made demountable to permit quick interchange of coil windings and quick conversion to permanent magnets. The pole-piece system used is unique in that the projection lens and objective-lens pole pieces have been incorporated into a single mechanical unit which can be quickly removed from the instrument for cleaning or for changing magnification. Figures 9 and 10 are photographs of an experimental pole-piece unit. In Fig. 10 one end has been disassembled to show its construction. Figure 11 shows a cross section of the pole-piece system used in the present model. It consists of eight accurately machined parts which are screwed together to form the complete unit. Axial alignment of all the parts is assured by precisely machined pilot and shoulder surfaces. The projection-lens system has a removable insert. With this insert in place the lens system has a maximum magnification of around 6000×. With the insert removed the system has a magnification of about 500×. As can be seen from Fig. 6, either the insert or the whole pole-piece system can be

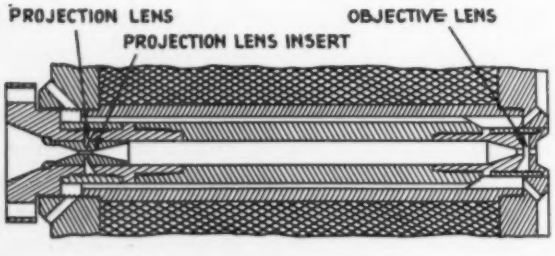

FIG. 11. A cross section of the most recent experimental pole-piece unit.

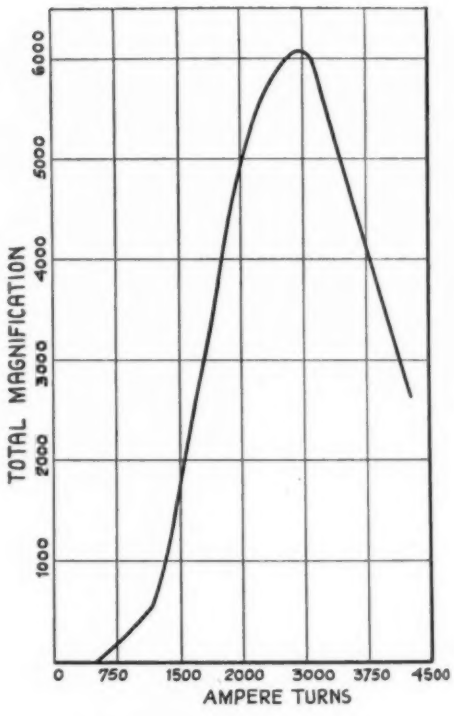

FIG. 12. A curve showing the relationship between the exciting current and instrumental magnification.

easily removed from the microscope by removing the fluorescent screen. While the instrument, as used, has two fixed magnifications it is possible, by varying the object-objective spacing and making a corresponding adjustment of exciting current, to adjust the magnification over a wide range. In Fig. 12 the magnification of the over-all system is plotted against the exciting current.

Because of the short distance between the projection-lens pole pieces and the final screen, distortion of the pin-cushion type was encountered in the image. It could be minimized though not completely eliminated by properly adjusting the spacing of the projection-lens pole pieces. Figure 13 shows the relationship between distortion of the final image and the pole-piece separation on the projection lens. This curve was obtained by photographing an object, first at the center of the field and second at the extreme corner of the field for a number of spacings. The magnification of the image was then measured accurately at each position. The minimum distortion was found to lie at the point where the

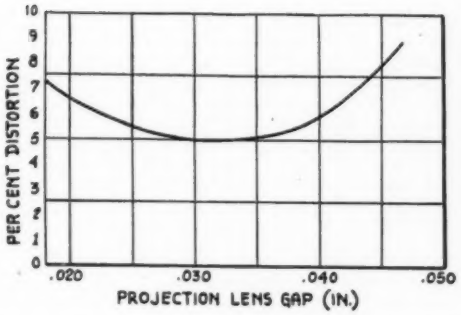

FIG. 13. A curve showing the relationship between image distortion and projection-lens pole-piece separation.

ratio of the projection-lens pole-piece separation to pole-piece opening was 1.19 : 1.

The instrument has also been tested using permanent magnets to supply the lens fields. There seems to be no serious drawback to this method. A magnification of 1500× was easily attainable and the resolution obtained was limited only by the photographic material. Figure 14 is a test micrograph of carbon black made with the experimental permanent-magnet lens system while Fig. 15 is a photograph of the microscope with the permanent magnets in place. The decision to use an electro-magnet was the result of a careful weighing of the factors involved in both types of construction. Actually the essential deciding factor was the complexity of design of the single current regulator required to control the electro-magnet as compared to the complexity of the various types of mechanical devices or high voltage controls required to focus a permanent-magnet lens system.

FIG. 14. A test electron micrograph of carbon black made with a permanent magnet lens system (24,000×).

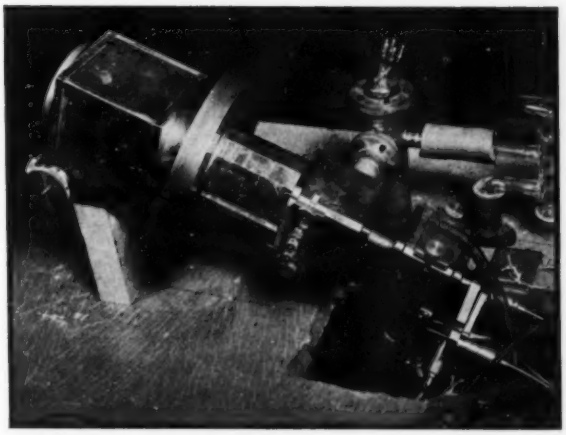

FIG. 15. A photograph of an early form of the small electron microscope in which permanent magnets were used.

With a transmission-viewed screen it was necessary to design an entirely new type of mechanism for inserting a photographic plate in front of the fluorescent screen and making the exposure. There appear to be innumerable ways in which this may be accomplished. In the device used, one external knob controls the entire cycle of operation for making a photographic record. In this mechanism the photographic plate is contained in a cassette resting in the bottom of the viewing chamber during instrument focusing. To make an exposure it is lifted into a position in front of the fluorescent screen by means of the external knob. Further manipulation of this knob actuates a catch so that the cassette and the plate are locked in position while the cover is released enabling it to be opened to make the exposure (see Fig. 6). This is also accomplished by the appropriate movement of the external knob. After the exposure is completed the reverse motion of the external knob replaces the cover, latches it, and lowers the whole cassette to its original position on the bottom of the photo-

graphic chamber. A door in the front of the instrument and immediately below the fluorescent screen can be opened for removing the cassette.

VACUUM SYSTEM

The entire microscope unit can be disassembled readily for servicing, the vacuum seal between units being maintained in the conventional way by synthetic rubber gaskets. Owing to the small volume of the instrument, airlocks are unnecessary but in order to avoid rapid decomposition of the diffusion-pump oil and in order to speed up the pumping cycle somewhat, a valve system is incorporated which enables the diffusion pump to be by-passed during the initial stages of pumping. High speed pumping is provided by a single-jet oil diffusion pump which was designed as an integral part of the microscope unit (Fig. 6). One notable feature of this pump is the fact that the tube which conducts the oil vapors from the boiler to the jet is double walled. The external wall provides physical support of the boiler and is connected to the latter. The inner wall forms a path for the pumping vapors and carries the pump jet. It is supported at its upper end by a thin-walled cup of low conductivity metal. This cup also seals off from the condensation chamber the space existing between the two walls. However, at its lower end this space is open so that it is filled with oil vapor from the boiler. It has been found that in this construction no oil is lost from the pumping stream by condensation on the walls of the conducting pipe. Moreover, there is less conduction from the jet and its support to the condensation chamber. A number of different types of jets were tested and one which has the form of a circular slit was found to have the

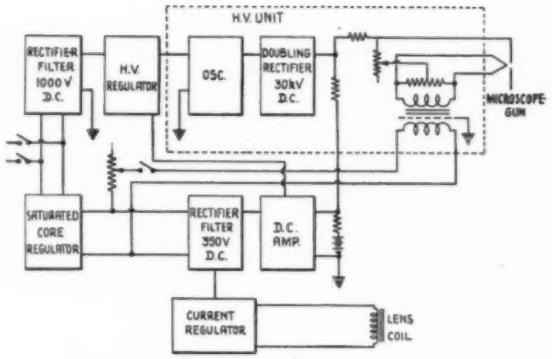

FIG. 16. A block diagram of the electrical circuits of the small electron microscope.

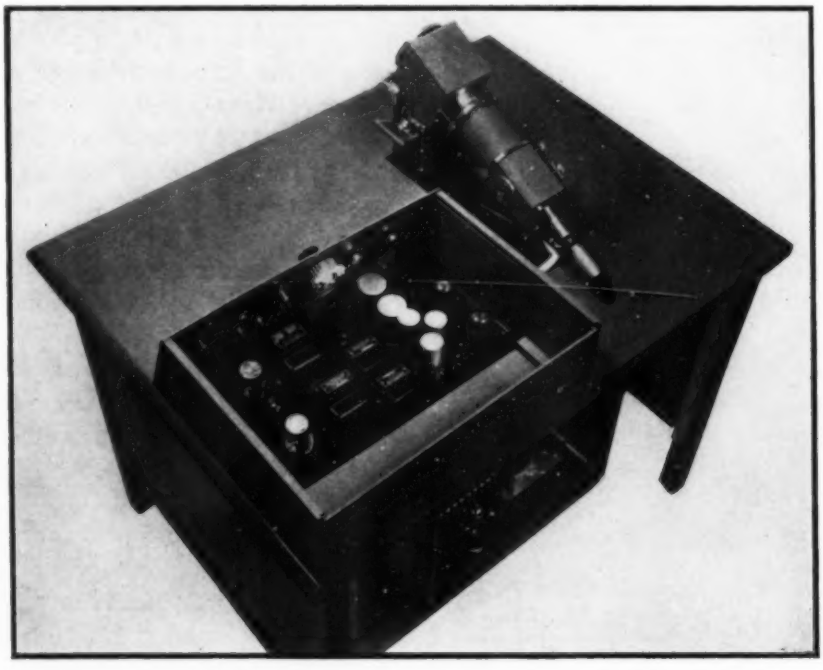

FIG. 17. A photograph of the low voltage chassis of the small electron microscope.

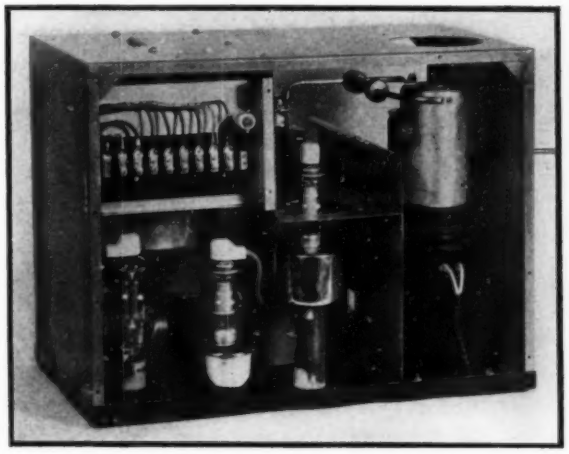

FIG. 18. A photograph of the high voltage unit of the small electron microscope.

highest pumping speed. In its final form this diffusion pump has a speed of around 10 liters per second and can reach an ultimate vacuum of 5×10^{-5} millimeter of mercury. The valve mechanism is contained within the block immediately below the photographic chamber. It consists of three properly connected valves using rubber gaskets as a sealing material. The valves are actuated by rods which are lifted by sloping cam surfaces on a single sliding cam plate. Steel helical springs supply the return motion while the vacuum seal is provided by the conventional packing-gland construction. The cam surfaces are so arranged in the cam plate that the complete pumping cycle is passed through by one to-and-fro-motion. The timing of the operation is left to the operator. A small fourth valve is provided as an air inlet and while it is manually operated it is interlocked in such a way that air cannot be admitted to the microscope when it is in operation. Similarly filament current and high voltage cannot be applied to the instrument except when the valve is in final operating position. The time required for pumping the instrument when a specimen or photographic plate is introduced varies between $1\frac{1}{2}$ and 3 minutes according to the length of time it has been left open.

POWER SUPPLIES FOR THE NEW INSTRUMENT

In the power supply which was designed by A. W. Vance, the more or less conventional design established in the Type B microscope[3] is followed. As a result of the elimination of the condenser lens and projection lens and the reduction of the accelerating voltage from 60 to 30 kilovolts, a large reduction in the size and complexity of the power supply was possible. The complete circuit has only 11 standard vacuum tubes. The power requirements were calculated for a magnification of 5000×, a resolving power of 20A, and an operation cycle time (from completion of focusing to end of exposure) of 30 seconds. These requirements are as follows:

1. An accelerating potential of 30 kilovolts (fixed) which does not vary more than 0.015 percent during the operation cycle. It must be capable of delivering 1 milliampere of current to the microscope filament.

2. A filament heating current—6 watts (variable) which must be supplied at 30 kilovolts negative with respect to ground. The maximum permissible variation over the operating cycle is 0.5 percent.

3. A grid potential of from 0 to 75 volts negative with respect to filament. The current drawn in this circuit is negligible. The maximum per-

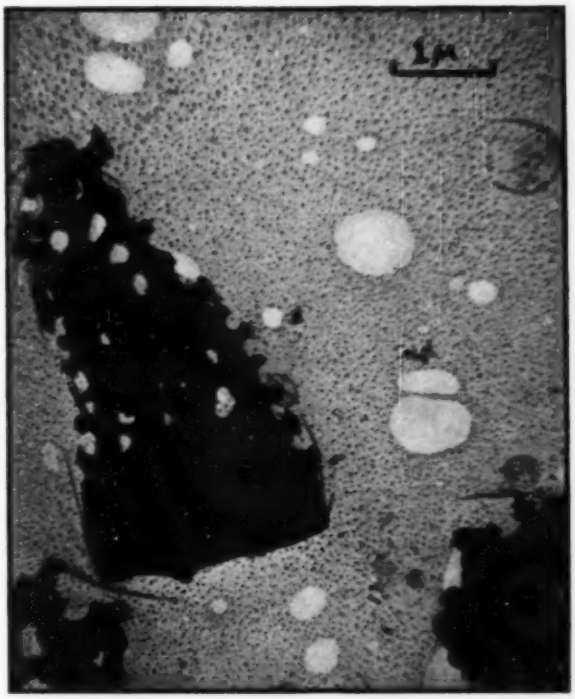

FIG. 19. Electron micrograph of diatomaceous earth on a membrane spattered with heavy metal; electronic magnification 3100× (13,000×).

missible variation over the operation cycle is 1 percent.

4. A coil current supply variable from 135 to 150 milliamperes direct current. Maximum permissible variation over the operation cycle is 0.007 percent. The adjustment must be continuous to within 0.007 percent.

Figure 16 is a block diagram of the complete power supply. It can be broken down into two physical units, the low voltage chassis and the high voltage unit. Figures 17 and 18 are photographs of these respective units. As can be seen, the low voltage chassis in this instrument is of about the same size as an ordinary home radio. The high voltage unit is air insulated and consists of a radiofrequency-operated doubling rectifier system which presents a series-resonant load to a self-excited driving oscillator. Regulation is obtained by a conventional negative-feedback circuit consisting of a stable voltage divider for obtaining the sample voltage, a two-stage direct current amplifier, and the regulator system which controls the plate input power of the driving oscillator. Dry batteries are used for the standard of comparison.

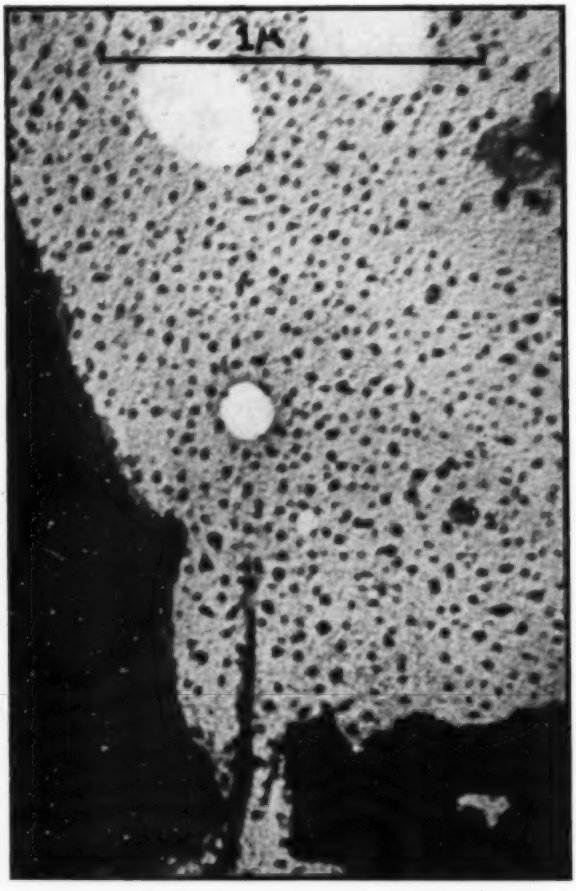

FIG. 20. A portion of Fig. 19 enlarged to 50,000×.

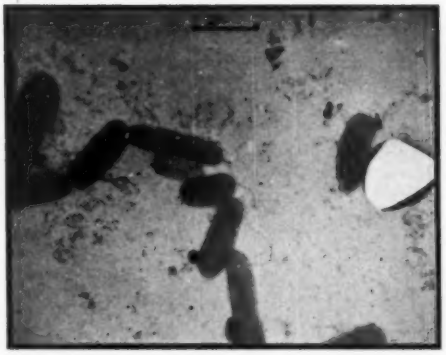

FIG. 21. Electron micrograph of some flagellated bacteria. Electronic magnification 6000×(8000×).

The microscope filament-heating power is obtained by means of a 60-cycle insulation transformer. This transformer was constructed with electrostatic shielding between primary and secondary windings. The filament heating is controlled by a variable resistor in the primary of the transformer. A line-voltage regulator of the saturated-core type is used to obtain the required stability in the filament heating. The voltage drop produced across a 100,000-ohm variable resistor by the microscope beam current provides the negative potential for the focusing aperture. In operation there was found to be a slight interaction between the focusing-aperture-potential adjustment and the best focus of the microscope. However, since the focusing aperture potential is not adjusted during the cycle of operation, this effect does not in any way disturb the final image.

Current for the coil is supplied by a conventional two-stage current regulator using a battery standard while two smooth controls are provided for the focusing adjustment. The finer of these gives a total variation of 2 percent in the coil current. The line voltage supplying this part of the circuit is regulated by a saturated core regulator. The vacuum measuring equipment consists simply of a thermocouple gauge operated

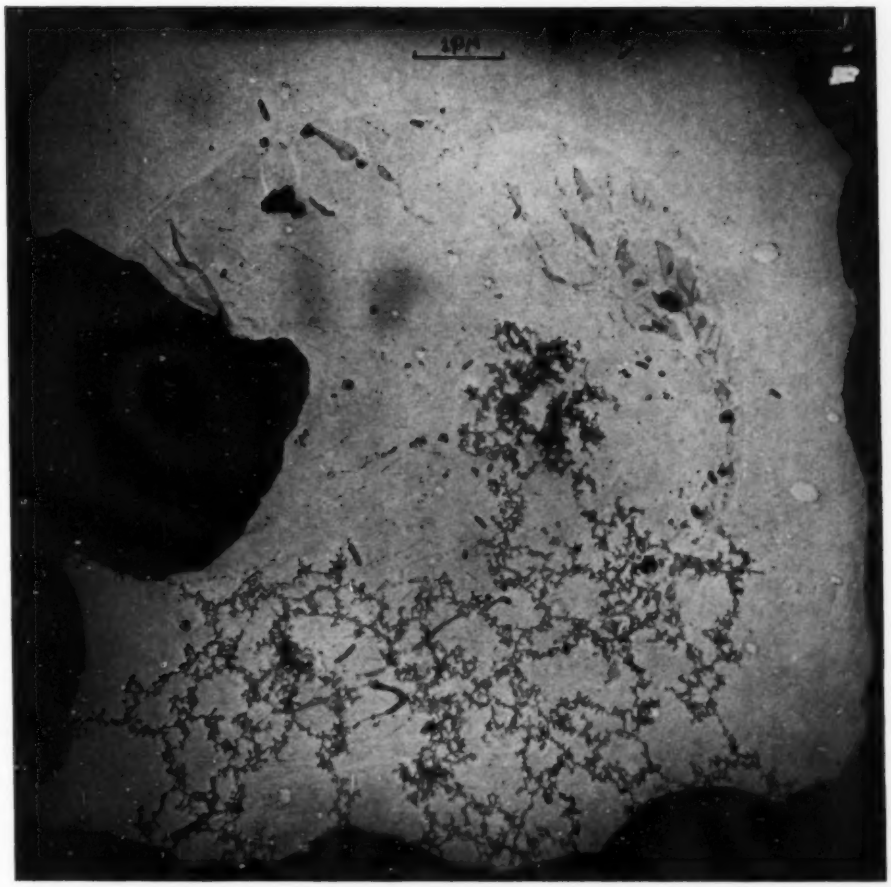

FIG. 22. A low magnification electron micrograph of carbon black and some bacteria. An entire single opening of a standard 200-mesh screen is imaged. Electronic magnification 500×(12,000×).

from a small step-down transformer and is also connected to the line-voltage regulator.

Only one small meter is used in conjunction with a multiple-position switch to indicate vacuum, beam current, coil current, and a number of other test voltages and currents.

A FEW RESULTS

Figures 19–22 show a few micrographs taken with this instrument. Figure 20 is an enlargement of part of Fig. 19 and clearly demonstrates the resolving power attainable. Figure 21 demonstrates the penetration that is obtainable in the majority of bacteriological specimens, while Fig. 22 shows a low magnification image.

ACKNOWLEDGMENTS

The authors wish to thank Mr. J. M. Morgan for his assistance in constructing and testing the power supplies.

Negative Resistance as a Machine Parameter

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(Received October 4, 1943)

A series generator in the operating range below maximum voltage possesses both d.c. negative resistance, $(-V/I)$, and incremental negative resistance, $(-dV/dI)$. It is shown that the incremental negative resistance is equal to $Kn-R$, where n is the speed, K the volts per r.p.m. per unit of field current, and R is the armature circuit resistance. Since the series generator has the incremental resistance property, it will support oscillations if the tuned circuit has the proper constants. It is shown that a generator load consisting of a separately excited motor can be represented by a condenser of sufficient size (in the order of farads), that oscillations at mechanically feasible frequencies of 0.2 to 1.5 cycles per second can be obtained. It is shown analytically that the magnitude of the capacitance varies directly with the moment of inertia of the motor armature and inversely with the square of the induction factor. It is shown experimentally that the frequency of oscillation of the circuit depends upon the motor induction factor and moment of inertia, and slightly upon the circuit resistance. The amplitude of oscillation is controlled by the circuit resistance.

INTRODUCTION

TWO useful definitions of negative resistance may be established. First, if the power P generated by a device is specified, then the negative resistance R_n is given by P/I^2 where I is the effective value of the current. Second, if the external characteristic (a plot of potential difference V as a function of current I), is specified, then the negative resistance r_n exists whenever V/I is negative, and is given by $r_n=-V/I$.[1]

The difference between these two definitions may be illustrated by considering a battery and a series generator. The external characteristic of the battery, Fig. 1(b), shows that the resistance R_n is positive only in the region in which the applied potential is less than the battery potential and has the same polarity as the latter. The resistance r_n is always negative, however. Hence a battery exhibits what may be called d.c. negative resistance R_n in the region $0<V<E_b$, but does not have what may be called incremental negative resistance r_n. For this reason a battery will supply power but will not support oscillations.

The external characteristic of a series generator, Fig. 2(b), shows that the resistance R_n may be either + or −, and that the resistance r_n is also + or − depending upon the region, as shown in Fig. 2. In the region where r_n is positive, 3 to 6, the series generator should be able to support oscillations.

SERIES GENERATOR OSCILLATOR

In Fig. 3(a) is shown a series generator A connected to a separately excited motor B. This circuit, familiarly known to many electrical

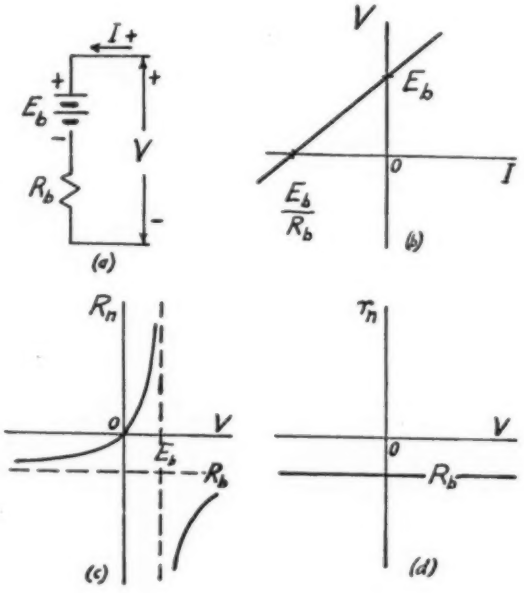

FIG. 1. Battery: (*a*) circuit, (*b*) external characteristic, (*c*) d.c. negative resistance R_n as a function of applied voltage V, (*d*) incremental negative resistance r_n as a function of applied voltage V.

[1] D. M. Tombs, Wireless Eng. **19**, 341 (1942).

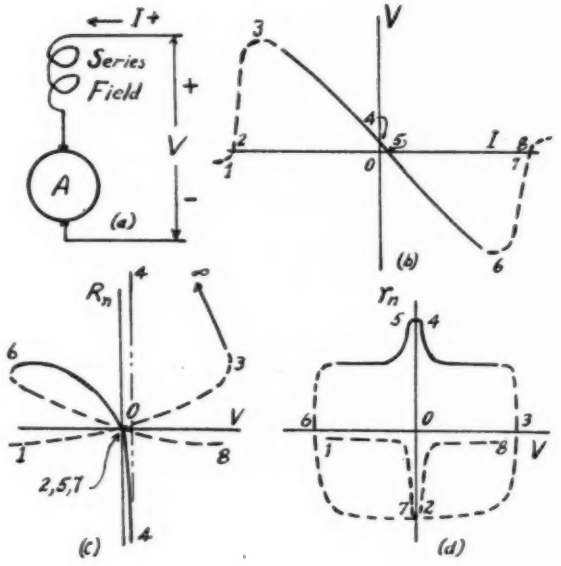

FIG. 2. Series generator: (*a*) circuit, (*b*) external characteristic, (*c*) d.c. negative resistance R_n as a function of applied voltage V, (*d*) incremental negative resistance r_n as a function of applied voltage V.

engineering students as the "bucking bronco," [2] will oscillate in such a way that the motor will continuously reverse direction because the generator current I is found to be alternating, if conditions are correct. The circuit contains negative resistance (provided operation is maintained in the region 3,6 of Fig. 2(d). The circuit likewise contains inductance, because of the field coil and the armature coils. The capacitance of the circuit may be shown to consist of the motor B and to be equal to $C=462\times10^{-6}J/(Z'\phi)^2$, where J is the moment of inertia of the motor armature, in lb. ft.^{2}, $(Z'\phi)$ is the induction factor of the motor expressed in volts per r.p.m., and C is expressed in farads. (See Appendix.)[3]

The circuit of Fig. 3(a) may be drawn in the form shown in Fig. 3(b). Oscillations of constant amplitude will occur when the resistance r_n is equal to R_0, the latter representing the electrical resistance as well as the equivalent effect of the friction losses in the circuit. The frequency of oscillation when the amplitude is constant is given by $f=1/(2\pi\sqrt{LC})$. Substituting the relation given above for C gives

$$f=(Z'\phi)/[2\pi(462\times10^{-6}JL)^{\frac{1}{2}}].$$

Hence the frequency of oscillation of the circuit should be directly upon $(Z'\phi)$ and inversely dependent upon $\sqrt{J}$. Figure 4 shows the relation between $(Z'\phi)$ of the motor and the frequency of oscillation of the circuit for two values of moment of inertia of the armature. The moments of inertia were only approximately determined from the dimensions of the machine.

The smaller value of J gave for C a value of 0.67 farad when $(Z'\phi)$ was 0.2. This very large value of capacitance is very striking, but it is to be remembered that the condenser does have associated with it a very large power loss and consequent high power factor.

The ratio between frequencies observed for the same value of $(Z'\phi)$ is seen to be about 1.15, which compares favorably with the square root of the ratio of the moments of inertia (1.26).

The curves of Fig. 4 should intersect at the origin, except for the fact that the losses in the motor are such that there will be no oscillations with a finite value of $(Z'\phi)$.

If the oscillations are stable, the effect of changing the resistance of the circuit should be simply to change the amplitude of oscillation,

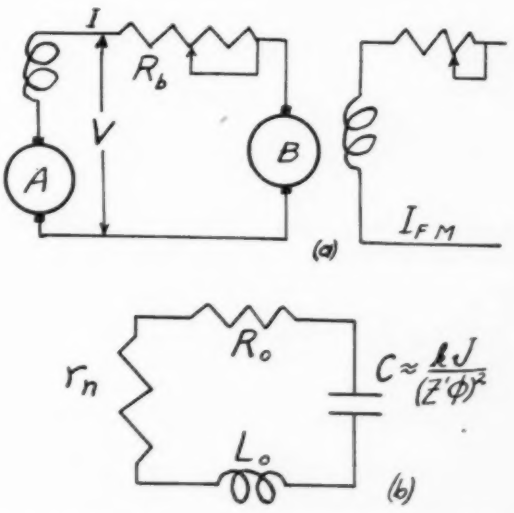

FIG. 3. (a) Series generator and separately excited motor, (b) equivalent circuit of (a).

[2] Cf. A. S. Langsdorf, *Principles of Direct Current Machinery* (McGraw-Hill, New York, 1940), 5th edition, p. 512. Kloeffler, Brenneman, and Kerchner, *Direct Current Machinery* (Macmillan, New York, 1938), 1st edition, p. 175.

[3] Cf. Alexanderson, Edwards, and Bowman, Gen. Elec. Rev. **43**, 105 (1940).

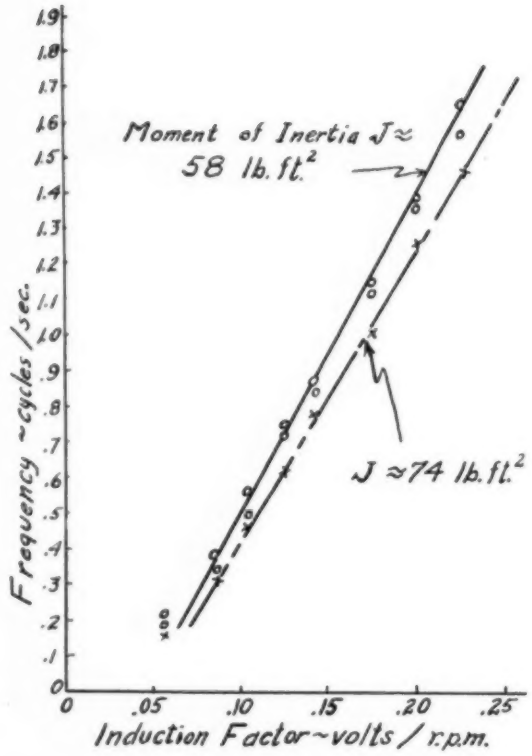

FIG. 4. Frequency of oscillation of motor as function of induction factor and moment of inertia of motor.

but not greatly to affect the frequency. In Fig. 5 is shown the effect of adjusting the resistance R_b of Fig. 3(a) on these factors.

Since it is a series oscillatory circuit, it is important that the series resistance be kept small. The power expended depends upon the current squared times the series resistance and the power supplied by the oscillator upon the current squared times r_n. The former must always exceed the latter for sustained oscillations. When R_b is too large, a direct current flows. The actual value of R_b cannot be determined because the variable current means a variable brush contact loss. The negative resistance of the circuit determined from Fig. 2(d) is about 0.5 ohm and hence the series resistance is about the same.

OSCILLATOR ANALYZED AS AN AMPLIFIER

A viewpoint which will be fruitful in other discussions of machines is to consider the series generator as an amplifier. Now it may be shown that there are three essential characteristics of an oscillating circuit; first, there must be an amplifier of sufficient gain; second, there must be arrangements made for feedback; third, there must be some method of controlling the phase of the feedback. A circuit having all three will oscillate if the feedback will be in such a phase as to maintain the output.[4] Furthermore, the oscillations will be sinusoidal in nature if inductance and capacitance are both present in the right amounts, and will be of the relaxation type if only one of these is appreciable.

The series generator satisfies all three of the requirements for oscillation. It is definitely an amplifier, since a small voltage applied to the series field will generate a large e.m.f. in the armature. Second, it has feedback if connected as a series generator, since the current in the armature may be caused to flow through the series field. The phase control is obtained by correctly connecting the series field, with respect to the residual magnetism. (This latter assumes that the frequency is so low that the flux and series field current are substantially in phase.)

The potential for the series generator is $V=IR\pm E_g$ where I is the current, R the armature circuit resistance, E_g is the generated e.m.f., which may be either + or − depending upon rotation direction and upon connections, and V is the applied e.m.f. But $E_g=(Z'\phi)n$ and, to a first approximation, $(Z'\phi)=KI$ if the magnetization curve is a straight line. Then $V=IR\pm KnI$ and the driving point impedance of the circuit is $Z_d=V/I=R\pm Kn$. If $Kn>R$ and the sign is negative (which means that the generated e.m.f. is in the same direction as the applied e.m.f.), the impedance is a negative resistance $R_n=r_n=Kn-R$.

Of principal interest is the fact that various values of negative resistance are obtained by a simple adjustment of the speed of the driving motor.

If the variations in the circuit take place quickly enough the self-inductance must be accounted for. The driving point impedance then becomes $Z_d=(R\pm Kn)+j\omega L$ but the same criterion for negative resistance holds.

If the flux is not in time phase with the current

[4] H. J. Reich, *Theory and Applications of Electron Tubes* (McGraw-Hill, New York, 1939), 1st edition, p. 323.

in the series field, the factor K above becomes a complex operator k_1-jk_2 and $Z_d=(R+k_1n)+j(\omega L-k_2n)$.

Oscillographic examination of the wave form of the oscillator described above shows that the oscillation is not of the relaxation type. However, when the field current of the motor is very large the changes in oscillating current become very abrupt, and it is possible to make the motor oscillate through an arc of less than 180 electrical degrees.

As an interesting sidelight, the oscillations provide an easy way for setting the brushes of the motor on the magnetic neutral. If the ammeter measuring the current I_{FM}, Fig. 3(a), is used as a guide, it will be noticed that when the brushes are not on the magnetic neutral this meter will experience a kick every time the current reverses. This inductive kick coincides with a rotation of the motor more in one direction than the other. This armature reaction can be used deliberately to unbalance the oscillations, in much the same way that a relaxation type oscillator vacuum tube circuit may be unbalanced by biasing the grid of one tube more than the other, or by adjusting the constants of the circuit.

It is proposed to continue this analysis further by examining other machines for instability and determining if negative resistance may not be a convenient way of explaining it.

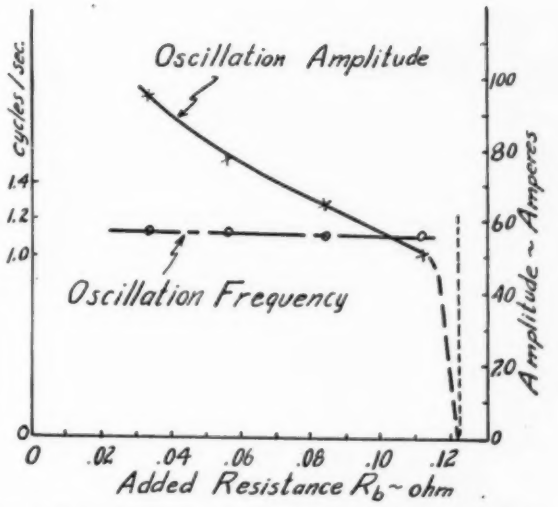

FIG. 5. Frequency of oscillation and amplitude of oscillation as functions of circuit series resistance.

APPENDIX

Neglecting electrical resistance of the motor and the friction and windage of the motor, an energy input to the motor results in an increase in kinetic energy of the motor. Stated mathematically, the energy balance may be written as $vidt=Jndn$ where v is the potential difference, i the current, n is the angular velocity of the motor, and dt and dn are increments of time and velocity, respectively. Since v may be written as $(Z'\phi)n$, idt as a change of charge dq, and the capacitance of a condenser is $C=dq/dv$, then $vdq=Jndv/(Z'\phi)$ and $dq/dv=C=Jn/v(Z'\phi)=J/(Z'\phi)^2$. Using lb. ft.^{2} to measure J and volts per r.p.m. to measure $Z'\phi$ results in the equation $C=462\times10^{-6}J/(Z'\phi)^2$ farads.

Magnification Calibration of the Electron Microscope

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A method is proposed for the magnification calibration of the electron microscope to compensate for the calibration errors caused by mechanical and electrical variations of the instrument. This method uses microscopic glass spheres of predetermined size, mounted directly on the specimen supporting film. A single calibrating sphere is then exposed on the same photographic plate with the sample. Any change, therefore, in the magnification of the specimen also causes a corresponding change in the magnification of the sphere, thus eliminating errors in calculating magnification arising from instrumental variations. The spheres are graded to a given size by controlled levigation and calibrated internally on a grating replica at a low magnification with a very low electron-beam intensity. By the use of low intensity of the beam, deformation of the replica of the grating is avoided. Also at the lower magnification many more lines of the grating replica may be included in the field for greater accuracy in determining the calibrating magnification. The spheres are placed on the specimen supporting film by evaporation from a water suspension. It is estimated that an accuracy of about ±3.0 percent is attained by the measurement of the image of the calibrated sphere on the same plate as the sample.

THE accuracy of all measurements made from electron micrographs depends of course on the accuracy of magnification calibration of the electron microscope. All of the methods of calibration in general use on the magnetic type of electron microscope have certain definite limitations of their own besides limitations inherent in the instrument itself. These instrumental errors alone allow an accuracy of only about ±10 percent in an object. The errors are caused by the inability to place the specimen supporting screen in the specimen holder and to seat this holder in the microscope stage in exactly the same calibrated position each time a different specimen is inserted. Variations in the specimen supporting film media and irregularities in the specimen screens also cause differences in the specimen position. These conditions require a change in the focal length of the objective lens in order to bring the specimen into focus, resulting in a change in magnification. Very slight fluctuations in the line voltage over comparatively long periods also cause a change in magnification. Other errors produced by the instrument itself are: lens hysteresis, aberrations, lens current and accelerating voltage changes, improper contact in the rheostat of the projector lens, and foreign particles in the lens pole pieces. Even taking these errors of the instrument into consideration by compensating for them as far as possible, often with considerable difficulty, not much better accuracy than the stated ±10 percent may be expected.

PRESENT METHODS OF CALIBRATION

Other limitations in the accuracy of calibration are to be found in the materials suggested for this purpose. Diatoms such as *pleurosigma obscurum* have been found to be too irregular in the spacing of the rows of puncta for calibration purposes. Also the lines of this diatom are so closely spaced that they may be counted only with difficulty even with the best optical system of a light microscope. Other materials or methods using the light microscope calibrated by a stage micrometer add the errors of that instrument and the micrometer to those of the electron microscope. Calibrating the microscope by the use of a large object of a calibrated size, having a finer structure, at a very low magnification and increasing the magnification in steps using the finer structures for the higher magnifications is rather tedious and tends to augment the over-all instrumental errors. Also the accuracy of the measurements for the higher magnifications is completely dependent on the accuracy attained in the successive measurements at the lower magnifications.

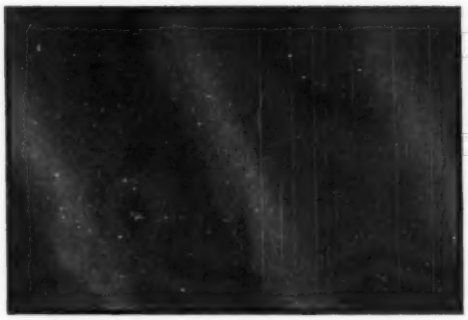

FIG. 1. 13,200×.

Another method of calibration uses a series of overlapping electron micrographs at high magnification of a large object of accurately calibrated size. Fitting together and measuring this composite picture is even more tedious than the preceding method since it must be repeated for many settings of the instrument with each specimen holder of different length. Also, as has been noticed, any relatively large movement of the stage requires refocusing of the object resulting in a slightly different magnification for each picture of the series, caused by irregularities in the stage bearing surfaces. Instrumental errors, as described above, are still present in all these methods of calibration. To these errors must also be added the errors of the micrometer or other device used to measure the calibrating object.

Perhaps the most widely used method of magnification calibration at present is the diffraction grating method.[1] This method has certain advantages, particularly the ease with which a calibration may be made at the lower magnifica-

[1] Charles J. Burton, R. Bowling Barnes, and T. G. Rochow, Ind. Eng. Chem. Ind. Ed. **34**, 1429–36 (1942).

tions. It has, however, certain inherent limitations such as the cost and difficulty of obtaining an accurately calibrated grating of at least 15,000 lines per inch. Formvar replicas[1] of such a grating which this laboratory has attempted to use have shown the individual rulings to be quite irregular in width and spacing, Fig. 1. At the higher magnifications the accuracy is very low because so few lines are included in the field. Errors are also introduced, particularly at the higher magnification, by the difficulty of selecting the exact points between which to measure since the replicas of the grating produce a fuzzy image of the rulings. This difficulty, however, may be minimized through the use of a recording densitometer, an expensive instrument to which relatively few laboratories have access. Other difficulties experienced with the Formvar replica of a grating are the unequal shrinkage and stretch over a large area by as much as 30 percent and the increasing opacity of the film to electrons. These troubles occur under the high intensity of electron bombardment necessary for focusing at the higher magnifications. Alone they would quickly destroy any advantage of accuracy of calibration by this method. Figures 2–4 at 1380 diameters of a Formvar replica grating on a single specimen screen illustrate this unequal shrinkage which is dependent upon the duration and intensity of electron bombardment. These difficulties may perhaps be overcome, at least in part, by the use of the polystyrene silica replica method described by Heidenreich and Peck.[2] Besides the difficulty of obtaining such a silica replica, it would very likely be necessary to destroy the calibrated metal grating in the process of separating it from the polystyrene replica, an expensive procedure for obtaining only one replica. The grating method as usually employed also has the instrumental errors with which to contend, unless the sample to be measured is placed either in or on the grating replica directly. Particles of many materials, particularly pigments, toners, and fillers cannot be properly dispersed for measurement by this method of mounting. Since the grating film must be thicker than the usual specimen supporting film, most of the small particles or structures of a sample would be obliterated due to the lack of contrast which is particularly severe in the thicker parts of the replica. With such relatively thick films trouble is also caused by chromatic aberration

FIG. 2. 1380×.

FIG. 3. 1380×.

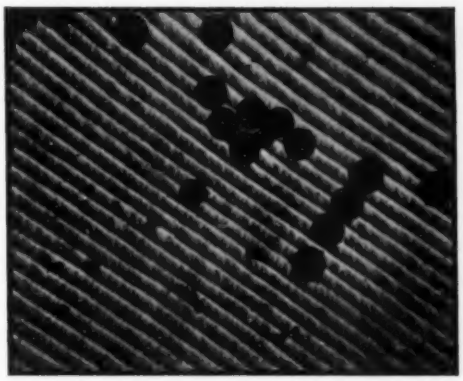

FIG. 4. 1380×.

[2] R. D. Heidenreich and V. G. Peck, J. App. Phys. **14**, 23–29 (1943).

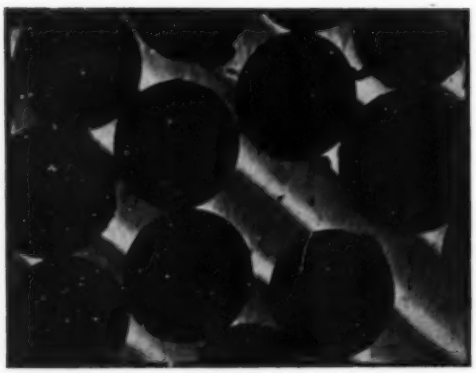

FIG. 5. 5300×.

due to the straggling of electron velocities in the film. These serious limitations make this grating method of magnification calibration of value only at the lowest magnifications where many lines may be included in the image field, very low electron beam intensities used, and where the instrumental errors do not need to be considered. Under these conditions the grating may be used as a primary standard.

In view of the existing methods of magnification calibration and their respective limitations a new method is suggested. Such a method makes use of microscopical spheres, Fig. 5, graded to a given size by controlled levigation. Having been calibrated internally on a grating replica, they are placed on the specimen supporting film from a water suspension. In the microscope a sphere is selected from among those of the largest size-group by the use of a millimeter or other scale ruled on the final image fluorescent screen. This calibrating sphere is then electron micrographed at either one end of the plate or to one side of the picture of the material to be measured. Due to the great depth of focus of the electron microscope (approximately five microns or more) both the sample in the nitrocellulose or Formvar film and the sphere on the film are in perfect focus. The magnification of each plate or even each exposure may thus readily be determined from the diameter of the calibrated sphere on it.

Glass was selected as the material from which the spheres were to be made. This material was chosen because it is easily pulverized in a steel ball mill and forms perfect, smooth surfaced spheres when the glass powder is blown through a blast burner of the fusing apparatus. Such glass spheres are easily dispersed in distilled water for mounting on a grating replica or the specimen supporting film.

MICROSCOPICAL SPHERE METHOD OF CALIBRATION

The spheres were made from pulverized Pyrex glass by a procedure modified from a method described in the literature.[3,4] These spheres which ranged in size from less than 0.25 micron to 5 microns were washed with concentrated nitric acid and dispersed with a mortar and pestle into distilled water. The spheres may be used directly by placing a small drop of the water-sphere suspension on the specimen supporting film and evaporating the water. However, it was found to be more advantageous to grade the spheres to a predetermined size by controlled levigation before using them. The procedure and apparatus for accomplishing this will be described in an article to be published by Dr. David Pall of these laboratories.

Upon removing the spheres from the levigation apparatus a number were mounted from a water suspension on a replica grating. At a very low magnification of the electron microscope, 412 spheres were electron micrographed on this grating. A size-frequency curve, Fig. 6, of the spheres was plotted from the measurement of these plates. This curve shows that the largest

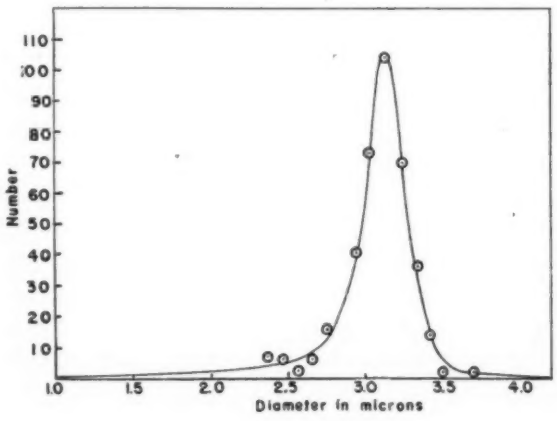

FIG. 6.

[3] K. Sollner, Ind. Eng. Chem. Anal. Ed. **11**, 48–9 (1939).
[4] C. R. Bloomsquist and A. Clarke, Ind. Eng. Chem. Anal. Ed. **12**, 61–2 (1940).

size-group consists of spheres 3.13 microns in diameter and that 79 percent of them are at or within 0.19 micron of this measured size. The magnification used in this case is calculated from the measurement of 42 grating lines in the same pictures as the spheres. These calibrated spheres, kept either in a water suspension (or dry), under sterile conditions, are mounted as needed by evaporating a small drop of a water suspension of them on the specimen supporting film. The images of 10 to 50 of them are measured on a negative or by a millimeter scale ruled on the fluorescent screen in the microscope, neglecting of course the ones that are obviously too large or too small. A single sphere, preferably an isolated one, is selected from that size-group having the largest number of spheres. This calibrating sphere is included in the micrograph of the sample at low magnifications. At the higher magnification it covers so much of the field that it is more advantageous to expose it at one end of the plate. For magnifications between about 20,000 and 30,000 diameters a few 0.5-micron spheres are added to the mount and one of these calibrated internally against the normal size sphere at medium magnification. This small sphere is then used in the same way at the highest magnifications as the larger spheres are used at the lower magnifications.

The grating method of calibration cannot be used directly at the higher magnifications for several reasons. The intense electron beam necessary for focusing distorts the replica, the lines are irregular in spacing, fuzzy, and very few are included in the field. However, at low magnifications of the electron microscope the beam intensity at any given point on the grating replica may be very much lower. This results in no distortion of the replica. The lines are sharp and many of them may be included in the image field for greater accuracy in measurement for determining the magnification. Under such conditions only may the grating method be used to advantage for the internal calibration of the spheres. These calibrated spheres are then used at all magnifications to eliminate the instrumental errors in the magnification calibration. The images of the spheres at all settings of the microscope are sharp and may easily be measured with a millimeter rule. The focusing on the spheres in the instrument is always an extremely critical operation particularly at the higher magnifications.

ACCURACY OF CALIBRATION

Since by this method each plate and even each exposure may contain a calibrating sphere, instrumental errors are completely eliminated. The errors that do occur are caused by the original accuracy of calibration of the grating and the calibration of the spheres from it. The internal calibration of the spheres on the grating may be accomplished with considerable accuracy provided certain precautions are taken. In stripping the Formvar replica from the calibrated grating a measured 0.7 percent shrinkage must be taken into consideration. Low beam intensities, possible only at the lowest magnification, must be used to eliminate changes in the replica. A sufficient number of grating lines, approximately 40, must be measured to minimize irregularities in the rulings of the grating and effects of field distortion. A sufficient number of uniformly graded spheres placed directly on the replica must be measured from the negatives produced. The images of the spheres on the negative may be measured with a magnifying lens containing a rule graduated to 0.1 mm. If these precautions are taken it is estimated that no greater total error than between ± 2 and 3 percent at most should occur at the lowest convenient magnification, i.e., approximately 1400 diameters of the electron microscope. This accuracy is possible only where instrumental errors are eliminated and a large calibrating object is measured since of course greater accuracy may be attained in measuring a large object than a small one. The high precision in calibration in itself is of no value unless the specimen to be measured is in the calibrated position. Such movement and time as are necessary in replacing the replica by the specimen will destroy the accuracy of calibration by introducing the instrumental errors previously described. Also the higher magnifications which will be necessary for most samples have not been calibrated.

USE OF THE SPHERES

In the measurement of a sample where the magnification of the electron micrograph must be accurately determined a water suspension of the spheres is placed on the specimen supporting film on a screen. The water is then evaporated, depositing the spheres on the film, and the specimen screen, with the adhering spheres, mounted in the microscope in the usual way. When it is seen by the electron microscope that the mount is suitable for measurement, the images of about 25 spheres are measured at random by a scale ruled on the final image fluorescent screen. The magnification used in this step is about 7000 diameters with the short focal length specimen holder. A sphere is selected of the most frequently occurring size and an exposure made of it at the desired magnification setting of the microscope. Exposures are then made of the sample in the immediate vicinity of the calibrating sphere. This precaution is taken to minimize any changes in the height of the specimen plane due to irregularities in the specimen stage bearing surfaces. The method of selecting the calibrating sphere is shown in the following table. Measurements are recorded of the images of the spheres, Table I, with the

TABLE I.

Run one Short focal length specimen holder		Run two Stereoscopic specimen holder	
Sphere image-size mm	Number of spheres	Sphere image-size mm	Number of spheres
21	1	18	1
22	1	18.5	6
22.5	0	19	20
33	7	19.5	11
23.5	9	20	4
24	22	20.5	1
24.5	6	21	1
25	3		

millimeter or other scale scratched in the center of the fluorescent screen. The most frequently occurring sphere image-size under any given set of conditions is selected in the microscope. For run one the sphere image diameter would be 24 millimeters and for run two, 19 millimeters. The microscope is then adjusted to the required magnification for the study of the sample. At such a setting the selected calibrating sphere is electron micrographed on the same plate with the image of the sample. Such calibration eliminates instrumental errors previously described.

Electrostatic charges which might possibly build up on the surface of the non-conducting glass spheres under electron impact do not apparently alter their size even under excessive electron bombardment. This lack of any change in the size of the spheres may be shown in the following way. The spheres, which have been graded to a predetermined size by controlled levigation, are checked first as to uniformity and size by the light microscope equipped with a Filar micrometer eyepiece. This eyepiece is calibrated directly by the steel grating from which the replicas are made. The spheres are then checked internally, in the electron microscope, against the accurately calibrated grating. This is done by placing them on a Formvar replica of the grating and taking electron micrographs of them at low and medium magnifications, under the full range of beam intensities. If any electrostatic charge does accumulate on the surface of the spheres under any intensity of electron bombardment, it does not alter their apparent size to any measurable degree, since their size is checked both by the superimposed grating and the calibrated light microscope.

OTHER USES OF THE SPHERES

The spheres may be used for checking image distortion at different points of the field. The spheres appear to be almost perfectly spherical when the microscope is in proper adjustment and the image of a sphere is in the center of the field, Fig. 7. However, the spheres themselves, although perfectly spherical, produce images that are slightly elliptical. The degree of ellipticity is dependent upon the microscope alignment, projector pole piece used, and the size of the hole in the first image screen. The extent and form of this distortion by the instrument at any given point in the field may readily be measured by moving a calibrated sphere to that point and electron micrographing it. Figures 8 and 9 illustrate this phenomena of ellipticity. Figure 9 shows the same spheres as Fig. 8 except that the object screen has been rotated approximately 90 degrees in a plane perpendicular to the axis of the microscope. One may see that the

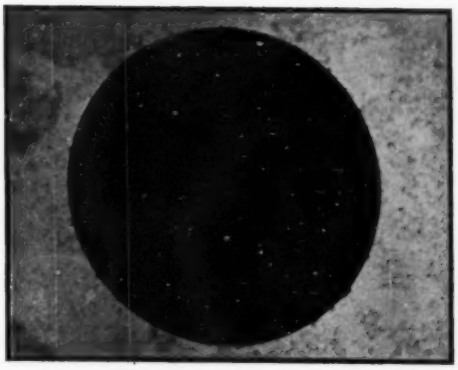

FIG. 7. 13,200×.

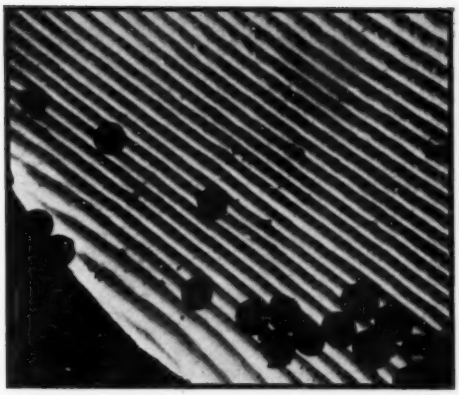

FIG. 8.

FIG. 9.

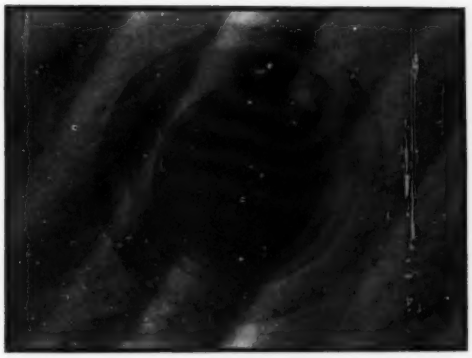

FIG. 10. 6900×–12,300×.

ellipticity in the same sphere in each picture is in the same position relative to the microscope axis. In measuring the image of the spheres for calibration an average diameter measurement is employed.

The spheres, besides being used for the calibration of each picture and the measuring of image distortion, are also used for making an approximate calibration curve of all instrument settings and specimen planes where instrumental errors do not need to be considered. For any given accelerating voltage and specimen holder a complete magnification calibration may be made on one plate by electron micrographing the calibrating sphere as shown in Fig. 10. In this picture the smallest image is 6900 diameters and the largest 12,300 diameters. The spheres are also used as a test object since they are so easily mounted, perfectly spherical, and produce sharp, clear-cut images when the microscope is in proper adjustment.

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Controlled Crystal Growth in Tantalum Ribbons*

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Microscopic examination of surfaces developed in tantalum ribbons on heat treatment in vacuum shows a dependency of surface structure on whether a.c. or d.c. supply is used for the heating current. Further modification of surface structure results due to recrystallization over a temperature range of 1900°K to 2500°K. Grain growth is shown to increase exponentially with the temperature. An optimum amount of cold working of the samples before heat treatment results in exaggerated grain growth yielding large single crystals.

I. INTRODUCTION

THE formation of a stable tantalum electronic surface to ensure reproducible data for work function determination or secondary emission involves periods of heating at high temperatures. The clean-up process, or evaporation of surface impurities and adsorbed gases, is only partly responsible for the production of a stable surface. Thermionic properties of several metals have been shown to be a function of the surface structure by Ahearn and Becker[1] *et al.* Recrystallization consequent upon heating affects all electronic properties which are a vector function of the surface crystal facets exposed.

It was shown by R. P. Johnson[2] that heat treatment of tungsten filaments in vacuum resulted in a modification of surface structure which varies with temperature, time, and manner of heating. Preliminary studies on other metals including tantalum filaments indicated results similar to those obtained for tungsten. Recrystallization of tungsten filaments consequent upon heating contribute by far the greatest surface modification. Grain growth in tungsten has been studied by R. B. Nelson,[3] M. H. Nichols,[4] and many others. The development of large chance grains in heated tantalum filaments was observed by D. B. Langmuir.[5]

The surface structure developed in tantalum ribbons shows with few modifications a similar surface structure dependence as was obtained for tungsten. Grain growth in the body of the samples as a function of the temperature follows an exponential law in essential agreement with results obtained for tungsten by C. S. Robinson.[6]

II. EXPERIMENTAL PROCEDURE

The samples used were ribbons of tantalum 2 mm wide and 15 cm long, cut from sheet metal 1, 3, and 5 mils thick. Polishing of samples was done with No. 1 Alumina flocculated in water. Surfaces polished for periods upward of 30 hours lost all initial die marks and appeared mirror smooth with occasional pits on the surface under a magnification of 500 diameters. The polished samples were mounted axially in a cylindrical tube. A 3-mil tungsten wire was mounted along the longitudinal axis of the ribbon and as close to it as the structure would permit, a distance of 4 to 5 mm. This arrangement permitted the pyrometric comparison of the brightness temperatures of the tungsten with the tantalum. The temperature of the tungsten filaments was obtained from the Jones-Langmuir temperature scale.[7] The etching of the samples was done with 3 parts sulfuric to 3 parts hydrofluoric acid in an equal volume of water. In this solution grain boundaries appeared on a polished sample in 60 to 90 seconds.

* Submitted to the Graduate School of the University of Notre Dame in partial fulfillment of the requirements for the Degree of Doctor of Philosophy.

[1] A. J. Ahearn and J. A. Becker, Phys. Rev. **54**, 448 (1938).

[2] R. P. Johnson, Phys. Rev. **54**, 459 (1938).

[3] R. B. Nelson, M.I.T. Physics Thesis (1938).

[4] M. H. Nichols, Phys. Rev. **57**, 297 (1940).

[5] D. B. Langmuir, Phys. Rev. **61**, 739A (1942).

[6] C. S. Robinson, J. App. Phys. **13**, 647 (1942).

[7] H. A. Jones and Irving Langmuir, Gen. Elec. Rev. **30**, 310 (1927).

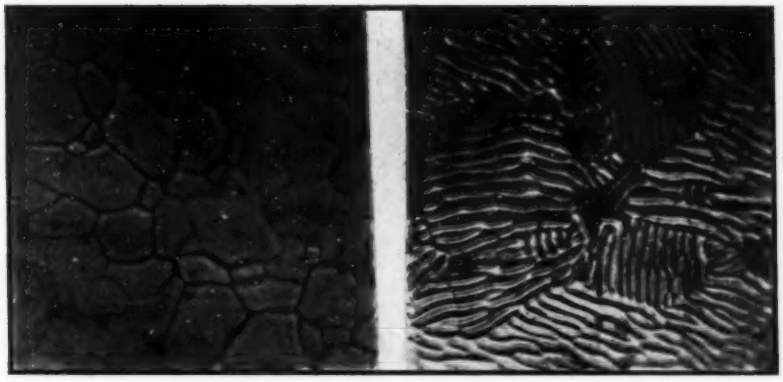

FIG. 1. (a) *Left:* Surface developed on a.c. heating (×375). (b) *Right:* Surface developed on d.c. heating (×375).

III. A.C. AND D.C. HEATING

Radically different surfaces are developed on prolonged heating in vacuum dependent on whether alternating- or direct-current heating source is used. Unpolished ribbons heated on a.c. for a period of 8 hours or more at 2400°K lose all initial surface irregularities and present a microscopically smooth surface with occasional grain boundaries visible with rounded edges, as illustrated in Fig. 1a. Prolonged heating further reveals the surface grain structure due to differential evaporation from the different exposed facets, yielding surfaces similar to those obtained on chemical etching. In general, however, the surfaces remain smooth with the exception of small regions near the ribbon supports where the surface structure resembles that obtained throughout the length of the sample on d.c. heating. Although observation was limited by magnifications up to 800 diameters, it is reasonable to assume that any further substructure that develops is on an atomic scale.

In samples heated on d.c. for periods up to 100 hours a characteristic rough surface develops visible to the naked eye. Microscopic examination reveals a step structure developed throughout the length of the sample. This structure is not associated with the original surface irregularities since it develops on both initially unpolished and polished samples. In the case of unpolished samples, irregularities are completely evaporated and this surface is superseded by the characteristic d.c. structure. Observations on samples of different grain size show that the step-like structure develops on a scale equal to the surface grain size of the metal. Each would-be-step runs the length of a grain. The contours developed are parallel on each grain with the directions orientated differently from one grain to the next (Fig. 1b). Heating up to 100 hours showed no further modification except to enhance this structure.

IV. EFFECT OF PLASTIC DEFORMATION

It was first noted by D. B. Langmuir[5] that heating of tantalum ribbons in vacuum at 2400°K for several hours often caused large grains to develop. Several such large grains of the order of 1 mm in section were studied by optical and x-ray methods showing that the surfaces developed in these occasional large grains were coincident with the low index atomic planes. In ribbons where the temperature was rapidly brought up over 1900°K a relatively uniform grain developed along the center of the ribbon with the large grains occurring at the edges. When the ribbons were annealed at 1600°K to 1700°K for 8 hours before advancing the temperature above 1900°K a uniform small structure developed throughout. The large grain structure could therefore be explained by the fact that strains introduced in shearing of the metal were responsible for the exaggerated growth at the edges.

The effect of strain upon the ultimate grain structure was determined by subjecting the

ribbons to stresses exceeding the elastic limit to produce plastic deformation. One-mil samples were strained by weighting the ribbon and by rolling in a preferred direction. Similar effects were produced in both processes with the exception that rolling in a preferred direction tended to produce a superficial small grained surface structure at low temperatures of recrystallization. This may be attributed to the fact that the surface grains were subjected to greater stresses than the grains in the body of the sample. At higher temperatures these grains were absorbed into the larger body structure.

Samples elongated from 1 percent to 10 percent of their original length were heated for a period of 7 hours at 2400°K; a sufficient time for an equilibrium structure to be established. The average grain size as a function of percent elongation is plotted in Fig. 2 on a semi-logarithmic scale. The points represent values obtained by averaging over the length of three samples. Readings at the ends of the samples were disregarded because of the uncertain grain size contrast caused by the steep temperature gradients at the ribbon supports. The optimum condition of plastic deformation for exaggerated crystal growth occurs between 2 percent and 3 percent. For elongations above 6 percent a slightly larger equilibrium structure than that of the unstrained ribbon results. Plastic deformation of 100 percent and 800 percent elongation did not show much deviation from the structure obtained at 6 percent to 10 percent.

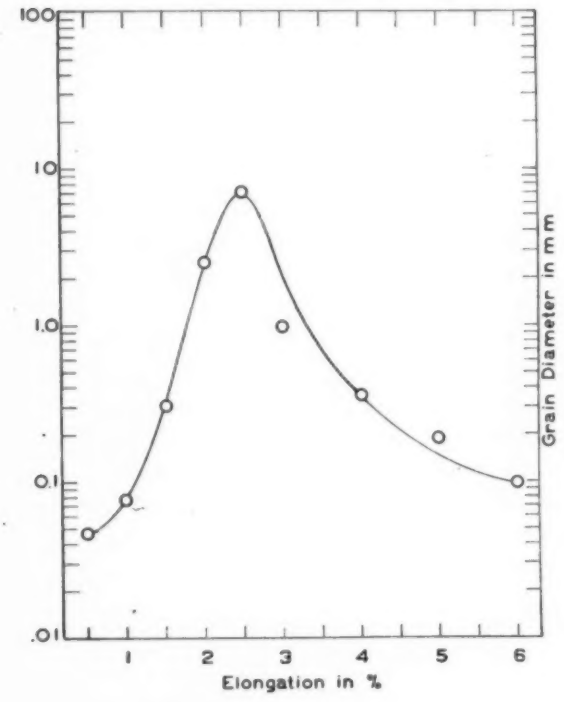

FIG. 2. Elongation in percent.

V. EFFECT OF TEMPERATURE ON GRAIN GROWTH

For a given previous history of a sample, the ultimate grain size obtained on recrystallization due to heating is a function of the temperature. It is very likely that recrystallization occurs at temperatures below 2000°K but inherent difficulties in microscopic studies limit practical observation to temperatures above that. The equilibrium structure reached at different temperatures is illustrated in Fig. 3. These represent average sections since in some cases exaggerated growth occurred that might be attributed to local temperature gradients or undue stresses in sections improperly annealed. The method used for ascertaining grain size was that first adopted by Jeffries.[8] It is evident from Fig. 3 that the resultant grain size increases rapidly with temperature; the grain size varying from 40 to 2 over a temperature rise of 500°K. Likewise the time required to reach equilibrium falls off from about 50 hours at 1900°K to less than 3 hours at 2500°K. Several 1-mil samples heated at temperatures above 2500°K indicated a decrease in final grain size but premature burnout did not permit systematic study.

To study the effect of grain growth with time at a constant temperature samples were heated from 1 to 10 hours at temperatures ranging from 1900°K to 2500°K. Results plotted in Fig. 4 show grain size as a function of the time of heating with the temperature as a parameter. Grain growth falls off exponentially as an equilibrium structure is approached. This result is compatible with the "Small Grain Hypothesis" for crystal growth first enunciated by R. B. Nelson.[3] In the process of growth a large nucleus grows at the expense of smaller adjacent grains by absorbing them. Several such chance nuclei

[8] Z. Jeffries, Met. Chem. Eng. **18**, 185 (1918).

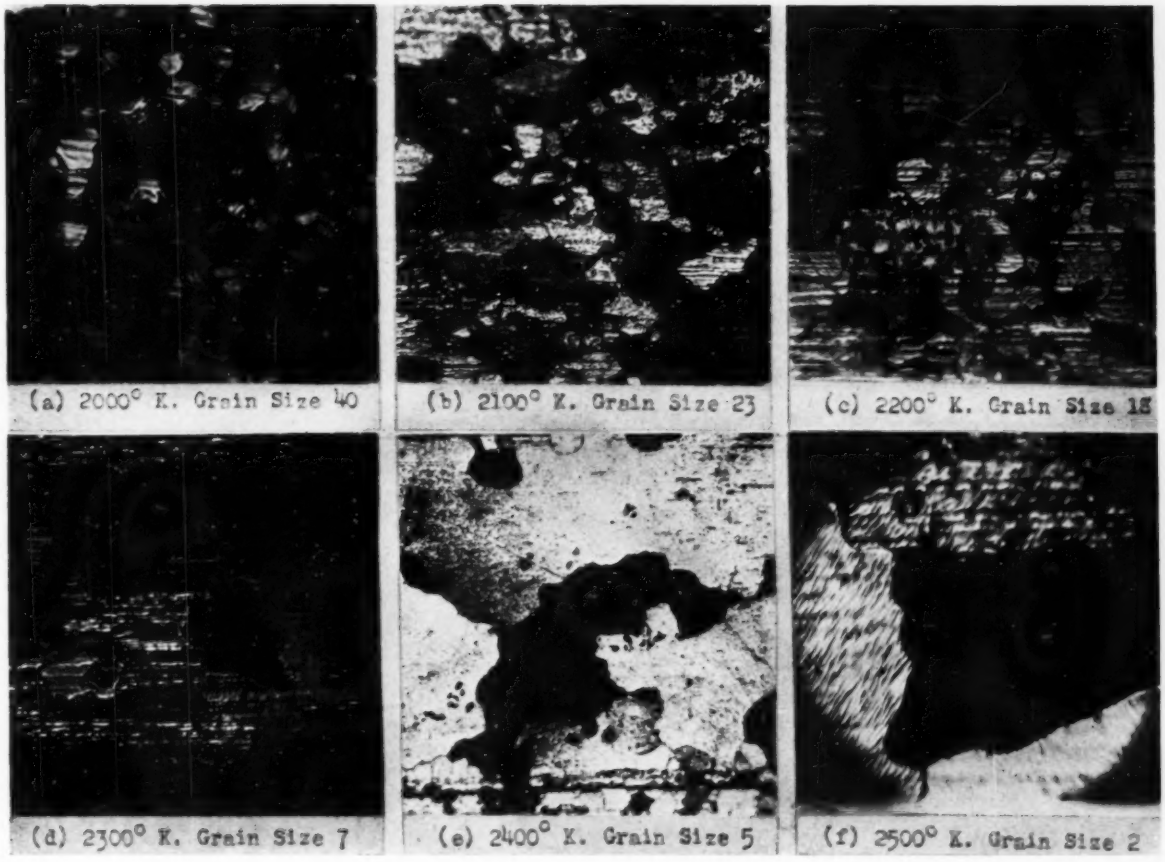

FIG. 3. (a) 2000°K. Grain Size 40. (b) 2100°K. Grain Size 23. (c) 2200°K. Grain Size 18. (d) 2300°K. Grain Size 7. (e) 2400°K. Grain Size 5. (f) 2500°K. Grain Size 2.

expand until the grains meet to form a common boundary. We would then expect a linear "free growth" with an asymptotic approach to an equilibrium state.

Figure 5 shows a plot of the slopes taken from the growth curves of Fig. 4 as a function of $1/T$. All points are in good agreement with a straight line indicating a temperature dependence of the form:

$$R = Ae^{-b/T}.$$

This equation was first applied to the rate of growth of crystals in tungsten wires by C. S. Robinson.[6] From the slope of the curve, b is estimated at 15,000°K as the activation energy for the process. Taking the value of R as 0.19 per hour from the curve at 2200°K and b at 15,000°K, the value of A is 1.1×10^{-5} per hour.

VI. DISCUSSION

The d.c. structure developed in the heat treated samples may be explained by the hypothesis introduced by A. J. Ahearn.[1] Since the probability of ion evaporation is negligible, the phenomenon must be attributed to positive ion surface migration in the electric field. As shown in experiments on migration of thorium over tungsten the rate of drift is a function of the underlying surface structure. Since different crystallographic directions present a different surface for the migrating tantalum atom cores we would expect the superimposed structure to be oriented with respect to the crystal lattice. A similar migration process would have to be assumed for surface diffusion down a temperature gradient, since the same type of structure develops at ribbon supports on a.c. heating. The

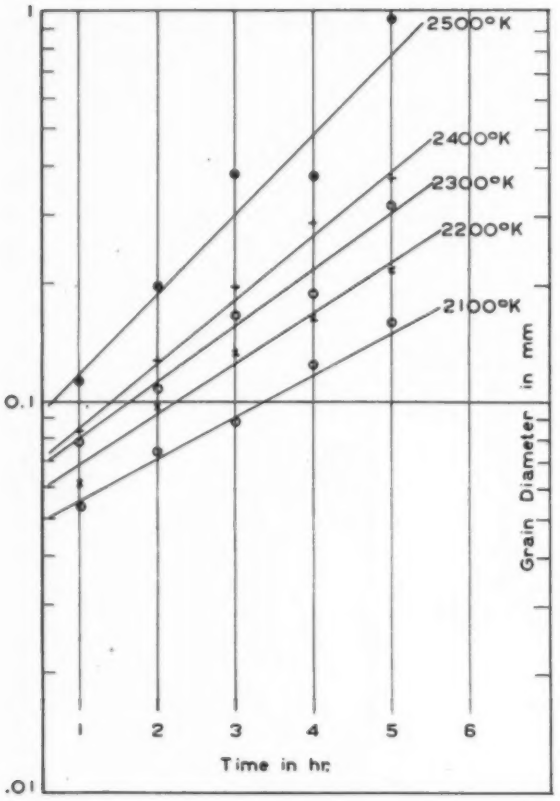

FIG. 4.

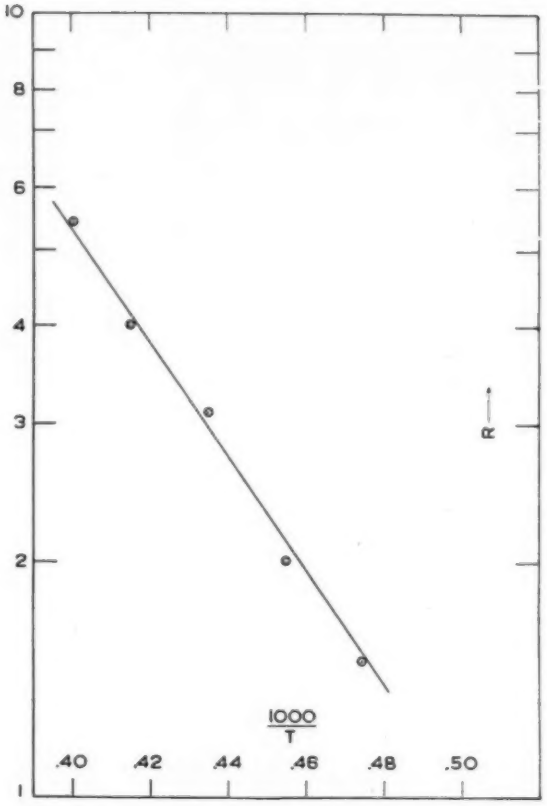

FIG. 5.

d.c. structure obtained follows in general the same pattern as that obtained for tungsten filaments by R. P. Johnson.[2] However, no superimposed structure transverse to the ribbon axis was observed in heating up to one hundred hours, which structure is reported by Johnson in tungsten filaments heated for periods up to 1000 hours.

The value of the activation energy for tantalum is about one-third of that obtained for tungsten by C. S. Robinson. This discrepancy is due chiefly to the assumption that growth to equilibrium in tantalum filaments which have a relatively small final grain structure is exponential. Had a linear free growth been assumed in the tantalum ribbons the values obtained would have been in good agreement with those obtained for tungsten, as one would expect for metals which are so similar in structure and physical properties.

Large single crystal grains filling the entire section of the ribbon and up to three centimeters in length were obtained by cold working and subsequent heating of samples. Straining of samples to the optimum amount, between 2 percent and 3 percent elongation followed by heating at a temperature between 2200°K and

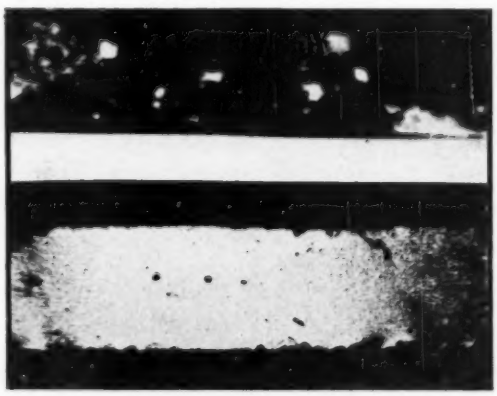

FIG. 6. (a) *Above:* Unstrained ribbon heated at 2400°K (×16). (b) *Below:* Strained ribbon heated at 2400°K (×16).

2400°K yielded exaggerated growth with large grains interspersed with small grain patches, as illustrated in Fig. 6a. Heating of samples under simulated temperature gradients yielded somewhat better results. Strained samples were tapered from 1 mm to 2 mm over a 15-cm length and heated by a slowly rising current over a period of two days in which the temperature rise was of the order of 200° to 300°K. Under these conditions a temperature gradient of about 20°/cm was maintained over the length of the sample. This method yielded 50 percent grains over 1 cm in length, as shown in Fig. 6b.

The author wishes to thank Dr. E. A. Coomes for his valuable assistance and helpful suggestions which made this work possible.

Studies in Lubrication

XII. Friction Behavior During the Slip Portion of the Stick-Slip Process

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The jerky form of motion described as "stick-slip," which is sometimes observed in the sliding of surfaces, has been studied by means of the stick-slip apparatus built in this laboratory. A comparative study of motion during the slip has been made for a number of combinations of unlubricated metals. The variation of friction with velocity has been determined for several typical cases. Most slip traces are symmetrical about their point of inflection indicating that the kinetic friction remains approximately constant. The static friction is greater, and a rapid drop to kinetic friction usually occurs. The kinetic friction is least at the end of slip, so that the friction-velocity relation is not reversible. There is evidence that the friction does not return immediately to its higher static value when the sliding surfaces come to rest. These results are considered in terms of several theories of friction.

INTRODUCTION

THIS investigation deals with sliding friction between metals in the absence of a lubricating film, so that the frictional behavior is determined by the properties of the metals themselves. Various proposals have been made concerning the nature of solid friction. The early investigators[2] attributed it to resistance to motion due to the interlocking of surface irregularities. More recently Tomlinson[3] has advanced a molecular theory according to which irreversible disturbances of molecules of the surfaces in contact are responsible, so that friction is entirely a surface phenomenon. Still more recently an interpretation, supported by their experiments, has been proposed by Merchant and Ernst,[4] as well as by Bowden[5] and several of his associates, to the effect that the bulk properties of the materials are of primary importance and that the frictional mechanism involves plastic deformation.

From experiments made on friction two conclusions may be drawn, namely, that it is extremely difficult to get reproducible results, and that friction is sensitive to a number of factors

[1] Now at Clark University, Worcester, Massachusetts.
[2] G. Amontons, *Memoires de Mathematique et de Physique* (1699), pages 259–286.
[3] G. A. Tomlinson, Phil. Mag. **7**, 905 (1929).
[4] Hans Merchant and M. Eugene Ernst, *Proceedings of the Summer Conference on Friction and Surface Finish*, Massachusetts Institute of Technology (June, 1940), page 76.
[5] F. P. Bowden and D. Tabor, Proc. Roy. Soc. **A169**, 391 (1939); F. P. Bowden and D. Tabor, Council for Scientific and Industrial Research, Melbourne, Australia, Bulletin No. 145 (1942); F. P. Bowden, A. J. W. Moore, and D. Tabor, J. App. Phys. **14**, 80 (1943).

which are difficult to control individually. For example, a very slight contamination of the surfaces greatly alters the friction. Such complications account for the difficulty in attaining reproducibility, since each measurement is a new experiment for which conditions may differ in some undetermined way from those of other measurements. Therefore, despite much interest and work, the knowledge of friction is quite speculative and it is possible that each of the effects described above may contribute to friction under particular conditions.

An important question for the understanding of friction and also the practical treatment of mechanical systems involving friction is its dependence upon velocity. Little is known about this question other than the general rule that static friction is greater than kinetic friction. Also the conclusion to be drawn from experiments on kinetic friction[6] is that it decreases as velocity increases, but that after a relatively small velocity is reached it remains almost constant. However, the interesting question as to just how the initial drop from static friction occurs—whether instantaneously or over a finite interval of time—remains obscure.

The general form of the friction-velocity characteristic may be predicted from the occurrence of stick-slip behavior. If one of two surfaces in frictional contact is driven slowly forward while the other is elastically fastened to a fixed position it is found that sliding is not continuous, but instead proceeds by a series of "sticks" and "slips." The latter surface sticks to the former and is dragged along with it until some limiting extension of the elastic support is reached, whereupon it is released abruptly and is drawn back. The exact nature of this jerky motion depends upon such mechanical conditions as the magnitude of the friction, the velocity of the driven surface, the elastic stiffness, and the inertia. For low friction and ordinary velocities, which are the conditions generally obtaining in the case of lubricated machinery, this behavior is not observed.

Blok[7] has shown that stick-slip motion may be a type of relaxation oscillation. He, as well as Kaidanovsky and Haykin,[8] has pointed out that such oscillations occur if initially the friction decreases as velocity increases. Moreover, if the motion becomes continuous when the velocity of drive is increased beyond some critical value the friction-velocity curve must reach a minimum and may thereafter bend upward.

The original apparatus used to study stick-slip phenomena was built by Bowden and Leben.[9] By means of such an apparatus Bowden and his associates have investigated a number of questions,[10] including the qualitative behavior of various combinations, the variation in conductivity during the cycle, measurements of average friction, and the effect of lubricants. They have considered the stick-slip behavior as a whole, but did not attempt to investigate kinetic friction during the slip. This is in line with their original interpretation of the phenomenon, according to which stick-slip is considered fundamental and as always occurring on a microscopic scale due to the clinging, elastic deformation, and shearing of minute surface irregularities, even though it may not be observable macroscopically. It is, of course, quite difficult to test directly the correctness of this theory, which attributes the characteristics of the measuring apparatus to the surfaces themselves.

Following the reasoning of Blok, the point of view was adopted in this investigation that a friction-velocity relationship of some significance existed, and an attempt was made to determine its form from a detailed analysis of the motion during slip. This was done with the purpose of contributing to the experimental knowledge of static and kinetic friction. The results are reported here. The limitations of the method and the difficulties encountered, many of these difficulties being characteristic of most friction experiments, are presented, since this may be of interest to other investigators.

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[6] See, for example, Galton and Westinghouse, Engineering **26**, 153 (1878).

[7] H. Blok, J. Soc. Aut. Eng. **46**, 54 (1940).

[8] N. L. Kaidanovsky and S. E. Haykin, Zeits. f. tech. Physik **3**, 91 (1933).

[9] F. P. Bowden and L. Leben, Proc. Roy. Soc. **A169**, 371 (1939).

[10] See references 4 and 8. Also see F. P. Bowden and L. Leben, Phil. Trans. Roy. Soc. London **A239**, 1 (1940).

APPARATUS

Since the stick-slip apparatus which was built in this laboratory has already been described,[11] only the main physical features will be recalled.

The motion of both surfaces is rotational. The steadily driven surface is a flat rectangular plate several centimeters in linear dimensions mounted about 12 cm from the center of a larger circular disk which is rotated by motor and gear drive. The linear speed of the driven plate is of the order of 0.02 cm/sec. A metal ribbon passes through a hole in the center of the disk and is held in position under considerable tension. The second surface is fastened to the ribbon by means of an arm. This surface is spherical, having a radius of curvature of approximately a centimeter. Thus the set-up is such that the second surface, which will be referred to as the slider, may vibrate torsionally, having a natural frequency of about 40 cycles/sec.

When the slider and plate are in contact, both the normal and tangential force are applied through the deflection of the ribbon spring. For a particular slip, or series of slips, the normal force is determined by measuring the pull perpendicular to the surfaces necessary to just separate slider and plate. The tangential force is determined by a calibration relating force and angular displacement. It should be mentioned here that there may be some variation in normal force, probably about 5 percent, due to deviations of the plate from flatness. The spring stiffness is such that for normal loads used, varying from 200 g to 1000 g, the slider is carried along several millimeters by the plate during the stick.

A photographic record of the motion is taken on a moving film. To record the position of the slider, a beam of light is reflected from a mirror mounted on the arm carrying the slider and is then focused to a spot on the photographic film. The magnification due to this optical lever is about 10, that is, if the slider is moved 1 mm the spot of light on the film is deflected about 1 cm. This deflection is in a direction perpendicular to the motion of the film. Consequently a continuous displacement-time curve is obtained for the slider, which will be referred to as a trace. The speed of the moving film is variable up to about 800 cm/sec. At this high speed a stretched-out trace is obtained of the motion during the slip.

By referring to the traces of Figs. 1 to 7 it will be seen that other information is also included. A zero line of the trace corresponding to zero displacement of the slider is obtained by reflection of another light beam from a fixed mirror. An oscillatory timing line is included, obtained by reflection from the mirror of a galvanometer driven by an oscillating circuit. The frequency of this is 1920/sec. Also several traces contain a record of velocity *versus* time. The desirability of recording such a velocity-time trace is discussed later.

Since the attempt to record the velocity directly gave unreliable results, this procedure was abandoned. Briefly, the method involved the use of a photoelectric cell whose illumination was made proportional to the displacement of the slider, a circuit to differentiate electrically the photo-current, and a galvanometer with mirror to measure the differentiated current. The photoelectric cell was illuminated by a light beam originating at a slit, the beam being intercepted by a knife-edge attached to the slider-arm. The knife-edge was inclined at an angle to the direction of the slit, and the system was so adjusted that the light intensity reaching the cell was proportional to the displacement of the slider. A d.c. amplifier to boost the current from the cell was necessary before differentiation. The differentiating circuit was similar in principle to that of Schmitt and Tolles.[12]

In addition, a record of the electrical conductivity was taken for a number of traces. In order to observe variations during the slip, which occurs in about 1/100 of a second, a galvanometer of high natural frequency and therefore low sensitivity was necessary.

No friction experiments can be done without considerable attention to the preparation of the surfaces. Probably there are about as many recommended ways of obtaining a clean surface as there have been experimenters. However, there seems to be agreement that the best pro-

[11] F. Morgan, M. Muskat, and D. W. Reed, J. App. Phys. **12**, 743 (1941).

[12] Otto H. Schmitt and Walter E. Tolles, Rev. Sci. Inst. **13**, 115 (1942).

cedure,[13] as judged by the criterion of reproducibility of results, is to expose a fresh surface by cutting or abrasion, and to perform the experiment as soon as possible after first removing abrasive or other loose material from the surface. To accomplish this the method adopted was first to wash the surfaces with tri-chloroethylene or acetone to remove excess grease, then to abrade with No. 400 emery paper, used dry, followed by the same emery paper used under running water, and then to wash with distilled water and allow surfaces to drain and dry naturally. This procedure gave as good or better reproducibility than any variations which were tried.

RESULTS

In Figs. 1 to 7 are reproduced a number of typical slip traces. D is the trace of displacement, V of velocity, and Z marks the zero line. The oscillating line is the timing line of period 1/1920 sec.

A few remarks about reproducibility are appropriate. The agreement among several experiments on a particular combination of metals depends somewhat on the combination. Two hard metals, such as steel on steel, give the least reproducible results. Nevertheless it is felt that the traces are of semi-quantitative significance in revealing frictional behavior. The same form of the trace for a given combination is generally obtained, while traces of different combinations may vary considerably. Probably not too much significance should be attached to a single trace, since considerable variations sometimes result. General similarity is usually found for most of the traces taken of a given combination. One of these may be chosen as typical, while another which deviates too radically may be disregarded. Although this procedure is not too satisfying, it can be justified on the basis that an unmeasured variation (contamination, lack of flatness of plate) sometimes interferes with a true observation.

As an illustration of the scattering of measurements obtained from such typical traces, Table I gives values for the coefficient of static friction μ_s from traces of Wood's metal on Babbitt. These traces were quite similar in visual appearance.

TABLE I. Coefficient of static friction for Wood's metal on Babbitt determined at several different loads.

Load in g	μ_s
200	1.25
250	1.33
325	1.04
325	1.19
350	1.01
Mean	1.17
Mean deviation	0.11

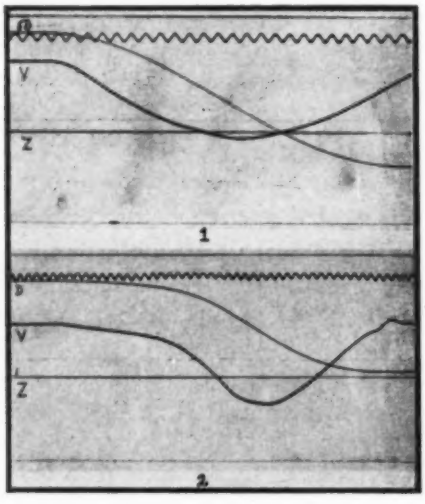

FIG. 1. High speed trace for SAE 52100 steel (Brinell 187) sliding on Babbitt (Brinell 24). Load: 400 g.
FIG. 2. High speed trace for SAE 52100 steel (Brinell 187) sliding on SAE 52100 steel. Load: 675 g.

The commonest type of trace, of which Figs. 1, 5, 6, and 7 are examples, exhibits a single slip from its initial displacement, the curve during slip being approximately symmetrical about its point of inflection. The distance which the slider slips varies from one combination to another, being considerable in the case of steel on Babbitt, Fig. 1, and very little in the case of brass on steel, Fig. 5. It is interesting that an interchange of the metal in the slider and plate often makes a great difference, as is illustrated by a comparison of the curve for brass on steel, Fig. 5, with that for steel on brass, Fig. 6. Figure 7, of brass on brass, illustrates that a similar behavior may be observed for a combination of two surfaces of the same metal, as for combinations of different metals.

[13] For example see paper cited under reference 4.

Several combinations give traces which depart from the simple symmetrical form. Copper on steel, Fig. 3, is characterized by a rapid initial drop followed by a gradual return to rest. The trace for steel on steel, Fig. 2, shows a slip which gets under way very gradually but drops more rapidly later. Wood's metal on Babbitt, Fig. 4, has a second slip in the reverse direction after first coming to rest.

ANALYSIS OF SLIP TRACES

In principle, the traces record enough information so that the frictional force at any instant of time during the slip can be found. Since the velocity at the same time can be found, it is possible to determine the friction-velocity relationship. In practice very precise records of displacement-time are necessary, and the attempts reported here have been only partially successful.

Since the velocity of drive of the plate is about 1/1000 of the average velocity during slip, it may be neglected. The slider during slip simply executes torsional oscillations subject to severe frictional damping. Therefore, the motion of the light spot reflected to the photograhpic film is of the same nature. Its equation of motion is

$$\ddot{y}+\omega_0^2 y=n(\dot{y}), \tag{1}$$

in which y is the displacement on the trace, $\ddot{y}=d^2y/dt^2$, ω_0 the angular frequency of free vibration, and $n(\dot{y})$ a damping term. The damping due to factors other than the friction between slider and plate is negligible, since the slider will execute many oscillations before coming to rest if it is allowed to oscillate without contact with the other surface. To a good approximation n is therefore proportional to the frictional force.

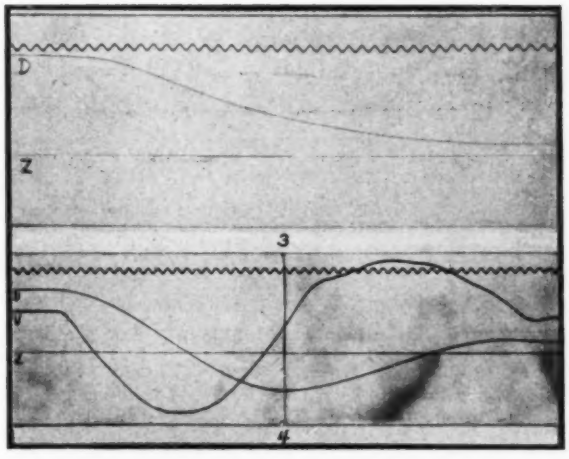

FIG. 3. High speed trace for copper (Brinell 70) sliding on SAE 52100 steel (Brinell 187). Load: 600 g.
FIG. 4. High speed trace for Wood's metal (Brinell 12) sliding on Babbitt (Brinell 24). Load: 225 g.

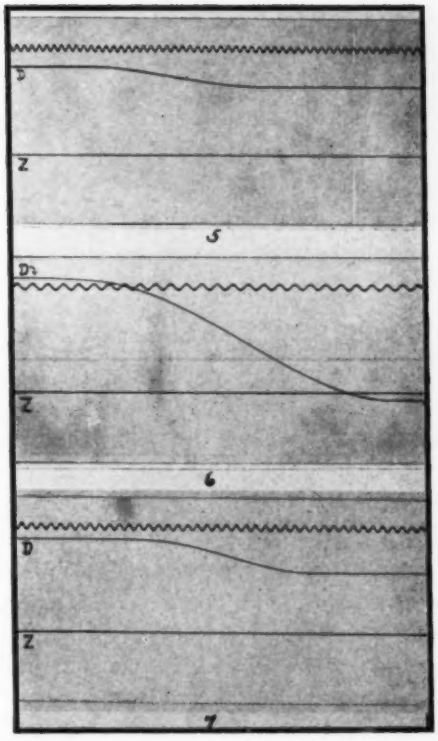

FIG. 5. High speed trace for brass (Brinell 119) sliding on SAE 52100 steel (Brinell 187). Load: 600 g.
FIG. 6. High speed trace for SAE 52100 steel (Brinell 187) sliding on brass (Brinell 119). Load: 1000 g.
FIG. 7. High speed trace for brass (Brinell 119) sliding on brass. Load: 950 g.

The static and kinetic friction may readily be determined. Let K designate the tangential force on the slider which will produce a displacement of the light spot on the trace of 1 cm. Let F_s designate the force of static friction, F_k the force of kinetic friction, and y_s the displacement just before slip. Then

$$F_s=Ky_s, \tag{2a}$$

$$F_k(\dot{y})=\frac{K}{\omega_0^2}n(\dot{y}). \tag{2b}$$

Equation (2a) is obvious from the definition of K and from the fact that when the plate velocity

is negligible, the static friction is equal to the restoring force of the spring just before slip. The proportionality factor connecting F and n, which appears in Eq. (2b), is readily determined by considering the static case or by reference to the equation of motion, Eq. (1).

The values of the constants were found to be:

$$K = 96.6 \text{ g/cm.}$$
$$\omega_0 = 249 \text{ radians/sec.}$$

The analysis therefore reduces to a determination of the acceleration of the position of the light spot on the trace. However, the precision of the observations is not great enough to permit this to be done directly. To find the acceleration, one may either twice differentiate the displacement-time trace or once differentiate the velocity-time trace. If the derivatives are calculated by the numerical process of taking differences, the errors of observations in the original data are considerably magnified by each differentiation. Therefore, it is desirable to obtain reliable traces of the velocity so that the acceleration may be obtained by a single differentiation.

Unfortunately, however, the velocity traces, which were obtained by the photoelectric method described above, exhibited spurious irregularities of the nature of peaks and sharp breaks in slope, as may be seen in Figs. 1 and 2, which do not appear to correspond to anything obtained from numerical or graphical differentiation of the displacement. A search was made for outside disturbances of the velocity-recording system but none was found. Since a velocity trace of a free vibration gave a regular sine wave, the irregularities described may have been due to transient effects in the system. Due to these difficulties the velocity method had to be abandoned. This necessitated that the displacement itself be measured with great precision. Now by directly reading the traces with a comparator the displacement on the trace may be found to within an error of somewhat less than 0.01 cm, giving a relative error of 1 percent or less. However, the relative error in the acceleration calculated from this is several hundred times as great, so that the scattering of points is of the same order of magnitude as the acceleration itself.

Consequently some method of smoothing the data had to be used. The actual procedure followed combined curve fitting with the smoothing process. A simple, analytic, form was first assumed for $n(\dot{y})$ such that the solution of the differential Eq. (1) was a curve similar to the displacement trace. This procedure has two advantages, namely, it shows how good a fit the simple interpretation gives, and it leaves only a small correction to n to be calculated afterwards. Thus, setting

$$y = y_0 + y_1, \quad n = n_0 + n_1, \tag{3}$$

where n_0 is the assumed simple function of $\dot{y}$, and y_0 is the displacement calculated from it, Eq. (1) gives

$$\ddot{y}_1 + \omega_0^2 y_1 = n_1(\dot{y}), \tag{4a}$$

$$\ddot{y}_0 + \omega_0^2 y_0 = n_0(\dot{y}). \tag{4b}$$

y_1 is the small difference between the experimental and the y_0 curves.

These points y_1 were plotted as a function of the time. The original irregularities in the data were much magnified on this scale. The method adopted for smoothing the data was simply that of drawing a smooth curve through the points for y_1. Integration of Eq. (4a) over a short time interval gives the following expression for the average value, $\bar{n}_1$, of n_1:

$$\bar{n}_1 = \frac{1}{t_2 - t_1}\left[\dot{y}_1(t=t_2) - \dot{y}_1(t=t_1) + \omega_0^2 \int_{t_1}^{t_2} y_1 dt\right]. \tag{5}$$

The smoothed curve for y_1 was used to determine graphically the slopes and the integral occurring in Eq. (5), so that the correction to n for the particular time interval was determined. As is true of any smoothing process, the results so calculated are not unique but depend on the particular form of the smoothed curve. However, a good estimate of the final form of the friction-velocity curve may be made if the smoothing is done in a physically reasonable manner.

The calculations for the following cases illustrate the application of this method.

I. Steel on Babbitt

The displacement trace for steel on Babbitt, Fig. 1, has the form common to most combinations of metals in that it is approximately sym-

metrical about its point of inflection. This type of trace may be fitted by the assumption of constant kinetic friction. Choosing the positive y axis in the direction of the displacement before slip, and setting

$$n_0 = \text{constant} = \omega_0^2 y_k, \tag{6}$$

the solution for a slider starting from rest at $y = y_s$ is

$$y_0 = y_k + (y_s - y_k) \cos(\omega_0 t + \Phi), \tag{7}$$

in which the phase Φ is determined by the condition that the argument of the cosine be zero at the beginning of slip. This solution is true only until $\omega_0 t + \Phi = \pi$, after which time the slider will remain attached to the plate, neglecting again the extremely small velocity of the plate. Equation (7) is that for simple harmonic motion of amplitude $y_s - y_k$ about the point of equilibrium $y = y_k$. The only effect of constant friction during the slip, therefore, is to shift the position of apparent equilibrium. The duration of the slip is one-half the period of free vibration of the system. From Eqs. (2b) and (6) the approximate value of the kinetic friction is

$$F_{k\ \text{approx}} = K y_k. \tag{8}$$

In fitting Eq. (7) to the experimental curve, y_s and y_k may be regarded as parameters to be chosen by least squares. This introduces no error

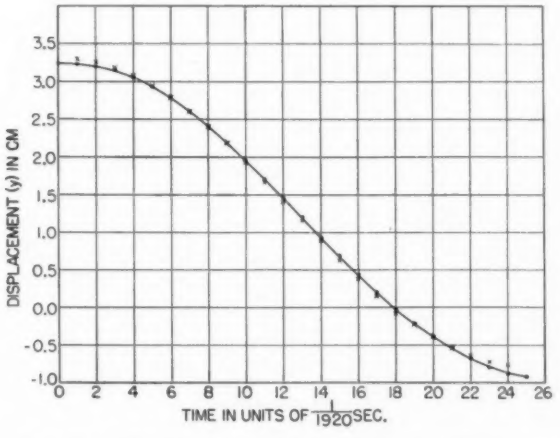

FIG. 8. Comparison of actual slip trace with that calculated on basis of constant kinetic friction, for steel sliding on Babbitt. Ordinates are displacement in cm measured on photographic trace. •: experimental points; x: points calculated by equation: $y = 1.26 + 2.03 \cos(\omega_0 t - 0.065)$; $\omega_0 = 249/\text{sec}$.

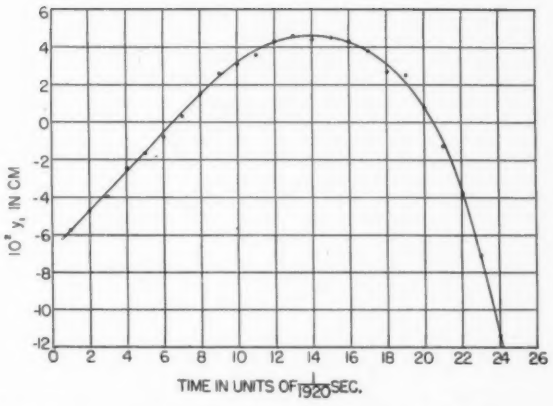

FIG. 9. Correction curves for y_1 for steel sliding on Babbitt.

in the final determination of the friction. Equations (4) and the calculations made therefrom hold true regardless of the values chosen for y_s and y_k, since the final solution is the sum of the solutions of Eqs. (4a) and (4b), and is independent of the manner in which this sum may be divided. However, the results are affected by an error in the determination of the instant when the motion begins, which, unfortunately, is difficult to ascertain. On the scale of abscissas of Fig. 8, the units of which are the period of the timing line (1/1920 sec.), this instant was chosen to be $t = \frac{1}{2}$.

Figure 8 shows the agreement obtained between the experimental curve and points calculated on the basis of constant kinetic friction. The assumed coefficient of kinetic friction is $\mu_0 = 0.304$.

Figure 9 gives the points y_1, which are the differences between experimental and calculated values, and shows the choice made for the smoothed curve drawn through these points.

The corrections to the friction were then determined from this curve by the use of Eq. (5). Figure 10 shows the final values for the coefficient of friction plotted against the absolute value of the velocity, which increases to a maximum and then decreases during the slip. Several facts are striking. The friction drops very rapidly from the static to the kinetic value—in fact, no intermediate value was found by this method. Also the friction does not retrace the same curve when the slider decelerates, but instead decreases somewhat.

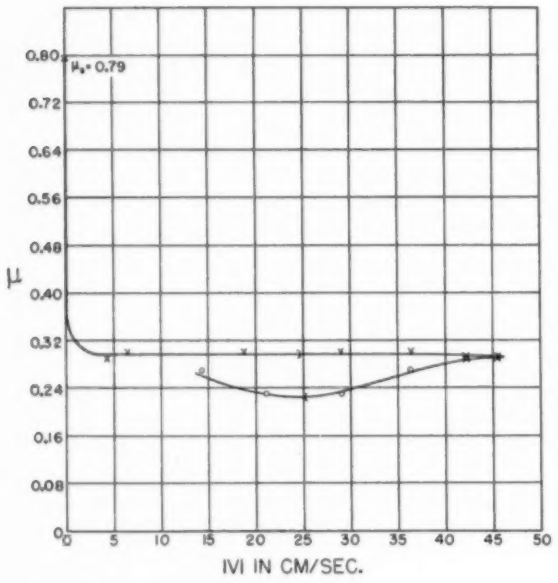

FIG. 10. Friction coefficient μ *vs.* velocity variation for steel sliding on Babbitt, as calculated from slip trace. x: $|v|$ increasing (slider accelerating); o: $|v|$ decreasing (slider decelerating).

II. Wood's Metal on Babbitt

This trace, Fig. 4, is typical of Wood's metal sliding on most metals and of tin on several metals. The slider is carried some distance beyond the equilibrium position during the first slip so that there is a second slip in the opposite direction. These two slips will be referred to simply as the first and second slip. As will be shown later, the second slip results from the high ratio of static to average kinetic friction. It is quite significant, because it shows the friction does not return to its initial high static value when the slider first comes to rest, for if this were so the second slip could not occur.

Each portion (before and after the minimum) of this trace may be approximated by a curve calculated on the basis of constant kinetic friction. As in the previous case the equation of motion and solution for the first slip are given, respectively, by Eqs. (6) and (7). During the second slip, since the friction is reversed,

$$n_0 = -\omega_0^2 y_k,$$

so that the equation of motion is

$$\ddot{y}+\omega_0^2(y_0+y_k)=0, \qquad (9)$$

for which the solution is

$$y_0 = -y_k+(y_s-3y_k)\cos(\omega_0 t+\Phi), \qquad (10)$$

Φ being such that the argument of the cosine is π at the beginning of the second slip.

Figure 11 shows the agreement between experimental and calculated curves. Two fits are shown. For the first, the parameters y_s and y_k are different for the two slips so that each slip is fit separately as well as possible although there is a discontinuity where they meet. The phases Φ for each slip were chosen to give the instant of beginning of slip coinciding with that for the experimental curve as well as could be determined. The second fit has one value of y_s, y_k, and Φ for both slips, with Φ so chosen that the minimum lies at the same position for calculated and experimental curves. The agreement is good for the fit to the first slip alone, but poor for the second slip, probably because this slip is completed in less than one-half the period of free vibration.

The correction to the friction was computed from the y_1's obtained from the separate fits. Figure 12 gives the final calculated coefficient of friction *versus* absolute velocity relation. The

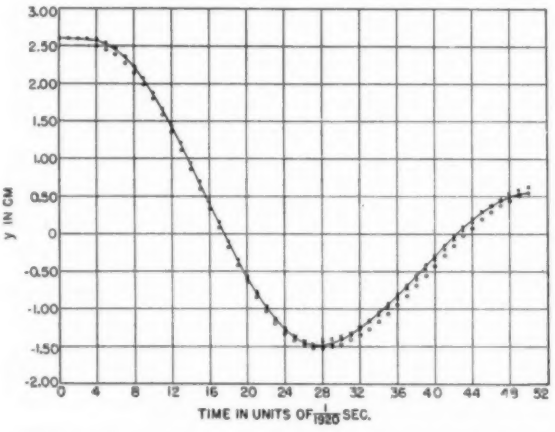

FIG. 11. Comparison of actual slip trace with that calculated on basis of constant kinetic friction for Wood's metal sliding on Babbitt. Ordinates are displacement in cm measured on photographic trace. •: experimental points. x: calculated points fitting each slip (half-period) separately, i.e., $y=0.550+2.035\cos(\omega_0 t-0.442)$ for first slip; $y=-0.384+1.038\cos(\omega_0 t-0.433)$ for second slip; phase chosen so that the time beginning of slip coincides with that of experimental curve. o: calculated points fitting whole curve at once, with phase chosen so that minimum coincides with that of experimental curve, i.e.: $y=0.484+1.965\cos(\omega_0 t-0.433)$ for first slip, and $y=-0.484+0.997\cos(\omega_0 t-0.433)$ for second slip, $\omega_0=249$/sec.

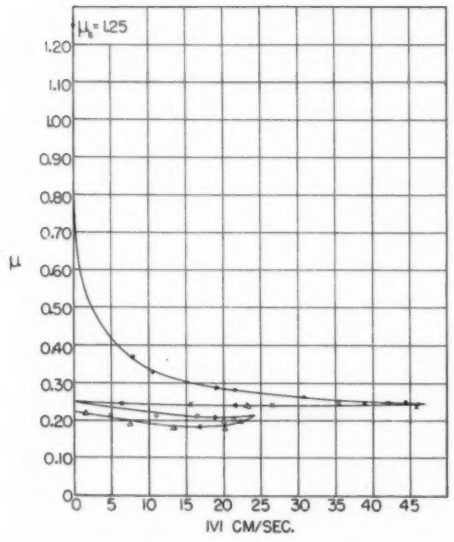

FIG. 12. Friction coefficient μ *vs.* velocity variation for Wood's metal sliding on Babbitt, as calculated from slip trace. •: slider accelerating, first slip; x: slider decelerating, first slip; o: slider accelerating, second slip; Δ: slider decelerating, second slip.

drop from static to kinetic, friction although rapid, does not appear to be instantaneous. Moreover, the friction does not return to its higher static value when the slider comes momentarily to rest between the two slips. The friction tends to decrease with time instead of showing a reversible dependence upon the absolute value of the velocity. In fact, the average friction is noticeably less for the second slip.

III. Steel on Steel

The curve analyzed (Fig. 2) shows an initially slowly accelerating motion followed by a more normal drop. The time of slip is longer than for previous cases. Traces obtained later indicate that this is not too typical of steel on steel and that usually the slip is more symmetrical and similar to the cases already discussed. However, the analysis is presented as an example of what is found from a curve of this shape.

A succesful fit was obtained on the assumption that the friction decreases linearly and moderately slowly from its static value during the early part of the motion, following which it remains constant. There are therefore two regions to consider. During the linear decrease the friction is

$$n=n_s-R|v|=n_s+R\dot{y}, \qquad (11)$$

$\dot{y}$ being negative, and R a constant. The equation of motion is

$$\ddot{y}-R\dot{y}+\omega_0^2 y=n_s. \qquad (12)$$

This is an equation of the type obtained for negative damping, for which the nature of the solution depends upon the values of R and ω_0. If $4\omega_0^2>R^2$ the solution is

$$y=A+Be^{\lambda t}\cos(\beta t+\Phi), \qquad (13)$$

in which A and B are constants of integration,

$$\lambda=\frac{R}{2},\quad \beta=(\omega_0^2-\lambda^2)^{\frac{1}{2}},\quad \Phi=\tan^{-1}\frac{\lambda}{\beta}+\theta,$$

and θ is a phase constant such that $\beta t+\theta=0$ at the beginning of slip. This curve was fitted to the first part of the experimental curve by adjusting the constants A, B, and λ. The instant of beginning of slip was taken to be at $t=1$ on the scale of abscissas of Fig. 13. During the time the friction remains constant, the solution is once again

$$y=A+B\cos(\omega_0 t+\Phi). \qquad (14)$$

This was fitted to the latter half of the experimental curve, by adjusting A and B, the phase

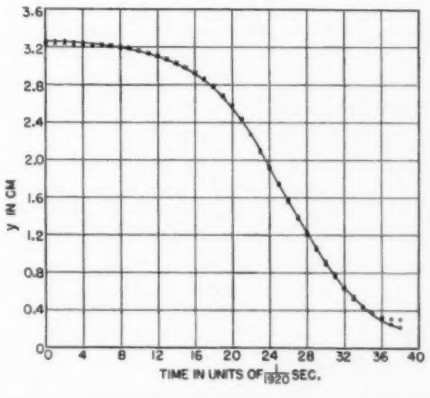

FIG. 13. Comparison of actual slip trace with that calculated on assumption of a linear decrease of the friction coefficient to a later constant value for steel sliding on steel. •: experimental points; x: points calculated on assumption of: $n=n_s-R|v|$, i.e.,

$$y=3.18+0.110e^{.111t}\cos(129t+0.891);$$

o: points calculated on assumption of constant kinetic friction, i.e.:

$$y=1.70+1.40\cos(\omega_0 t-1.680),$$

$\omega_0=249$/sec. and the phase chosen for exact fit of point of inflection.

Φ having been chosen so that the point of inflection coincides with the experimental curve.

The good agreement obtained, shown in Fig. 13, may not be surprising considering the number of parameters. The final friction-velocity curve, shown in Fig. 14, deviates little from the assumed

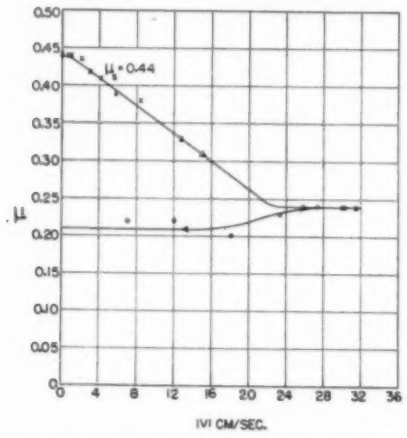

FIG. 14. Friction coefficient μ *vs.* velocity variation for steel sliding on steel, as calculated from slip trace. x: $|v|$ increasing; o: $|v|$ decreasing.

form. Once again it is evident that the friction does not reverse itself during deceleration, but instead continues to decrease somewhat.

DISCUSSION

The above analysis has indicated the nature of frictional behavior during the slip. The dependence on velocity is not simple and reversible, and in general the friction is less at the end of slip than at the beginning. The approximation of a sharp drop from static to constant kinetic friction is good for symmetrical traces. For traces for which the slip begins more slowly, as in the case of steel on steel, the friction initially drops more gradually. Probably an analysis of the trace for copper on steel, Fig. 3, which has a long gradual drop during the latter part of the slip, would show a steady decrease in friction throughout the slip cycle. The magnitude of the static and average kinetic friction vary considerably from one combination to another.

Figures 15 to 19 contain reproductions of several other typical slips among the various combinations of metals studied in this investigation. On these traces D is the displacement, Z the zero displacement, V the velocity, and C the electrical conductivity.

An interesting question one may ask is whether the friction returns immediately to its static value when the slider returns to rest. All the evidence thus far cited indicates that it does not. It certainly does not in the case of Wood's metal on Babbitt, for if it did motion would be arrested at the end of the first slip. This is true for all other combinations for which a second slip is observed, which include Wood's metal on all metals tried, and tin on several metals. For these combinations the ratio of μ_s/μ_k is large.

One can determine the critical value of μ_s/μ_k at which second slips should occur if it is assumed that the friction remains at a constant value equal to the kinetic friction, even when the slider returns to rest. This frictional force, according to Eq. (8), is Ky_k. A second slip occurs if the restoring force of the spring is greater. From the definition of K the magnitude of this force is $-Ky_f$ if y_f is the value of y at the end of the first slip, which is negative. Therefore the condition necessary for a second slip to occur is

$$-Ky_f > Ky_k.$$

From Eq. (7), $y_f = -(y_s - 2y_k)$, so that the above becomes[14]

$$y_s/y_k > 3,$$

or

$$\mu_s/\mu_k > 3, \tag{14}$$

since

$$y_s/y_k = F_s/F_k = \mu_s/\mu_k.$$

If a combination of metals for which μ_s/μ_k exceeded 3 showed no second slip one could conclude that for this case the friction returns to a higher value when the slider comes to rest so that the motion is arrested. However, no such case has been observed. In fact, a second slip occurred for Wood's metal on steel for a ratio slightly less than 3, which was probably due to

[14] This type of relationship—assuming constant kinetic friction—may be generalized to cases in which the slider may execute j complete damped oscillations and finally stick to the plate in the subsequent ith "quadrant," the latter being defined as a quarter cycle bounded by the zero line and a maximum or minimum amplitude. In terms of this notation, one finds that:

$$4j+i \leqslant \mu_s/\mu_k \leqslant 4j+i+1.$$

the decrease in friction at the end of the first slip. Thus the evidence indicates that the friction does not return to its static value instantaneously when the surfaces come to rest, but instead that it depends upon how long the surfaces remain at rest. Further verification of this is given by the higher static friction often observed for the first slip of a series when the surfaces remain in static contact a much longer time before the first slip than between succeeding slips.

The results reported above show that a friction-velocity relation has rather little meaning during sliding between metals which are in contact over only a limited area. It can be said that the static friction is greater than the kinetic if one restricts static friction to mean that between surfaces which have been in static contact for some finite time. The friction is not determined by the velocity alone, but rather by the past history of the motion.

This conclusion is not altogether surprising. It has long been known that friction produces wear, so that the surfaces must suffer severe treatment during sliding, especially when they are not lubricated. The pressures at the local points of contact must be high, as has been emphasized by several investigators.[4, 5] Visual observation of the plate and slider after being used in stick-slip reveals either a groove formed in the plate when the plate is softer, or metal from the slider wiped onto the plate when the plate is harder, or a combination of the two effects. It is significant that the disturbance of the surfaces is greatest at the beginning of slip. As already stated, the nature of slip is usually changed when the metal in the slider and plate are reversed. One could conclude from these facts that friction depends upon the condition of the surfaces and the mechanical properties of the metal near the surface, which in turn depend upon the history of the past motion.

This conclusion supports the bulk properties and plasticity explanation of friction rather than other theories. A purely molecular theory, besides giving an effect too superficial to account for the depth of deformation, fails to explain the history dependence. On the other hand, it is evident that the bulk mechanical properties of the metals are involved, and that plastic deformation occurs. Such an interpretation can also be applied to advantage to account for the history dependence.

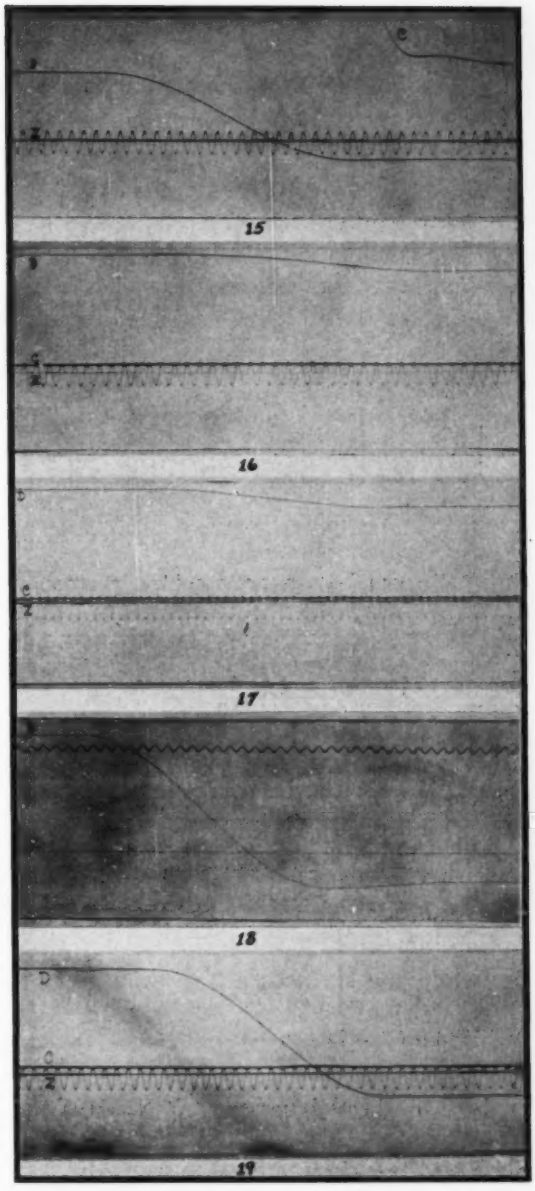

FIG. 15. High speed trace for Wood's metal (Brinell 12) sliding on Wood's metal.
FIG. 16. High speed trace for 17st aluminum (Brinell 116) sliding on aluminum. Load: 425 g.
FIG. 17. High speed trace for brass (Brinell 119) sliding on 17st aluminum (Brinell 116). Load: 525 g.
FIG. 18. High speed trace for tin (Brinell 6) sliding on SAE 52100 steel (Brinell 187). Load: 425 g.
FIG. 19. High speed trace for Babbitt (Brinell 24) sliding on Type 304 stainless steel (Brinell 143). Load: 350 g.

Bowden, Moore, and Tabor[5] have suggested that there are two important mechanisms responsible for sliding friction:

1. "Shearing" of adhesive metal-to-metal junctions formed at the local points of contact.

2. Resistance encountered due to high points of one surface plastically "ploughing" through the other.

This is a clarifying classification of the processes involved, even though there may be no absolute distinction between the two types. However, they are fundamentally different if "shearing" is understood to mean a discontinuous process of breaking junction, whereas "ploughing" is understood to involve continuous applied pressure and continuous plastic flow whether or not a furrow be formed. "Flowing" seems to be a truer description of the latter process, but in accord with the existing terminology, it is here referred to as "ploughing."

This shearing-ploughing theory fits in well with the results reported, and in addition the results support an extension of the theory. Bowden, Moore, and Tabor did not distinguish between static and kinetic friction. It seems probable that shearing occurs at the beginning of sliding and is responsible for the excess of static friction over kinetic, whereas ploughing contributes to both. The following qualitative explanation may then be made. Time is required for local adhesive junctions to form, probably because plastic deformation and rearrangement of surface atoms must first take place. After the junctions are sheared adhesion forces will be lower while the surfaces are in motion so that mainly ploughing takes place and the friction drops to its kinetic value. It does not return to its static value unless the surfaces remain at rest long enough for new adhesive junctions to be formed. The surfaces are torn during shearing so that the kinetic friction is greater at the beginning of slip and decreases somewhat as the irregularities are ploughed away.

It should be pointed out that molecular forces or other possible causes of friction are not excluded and may be operative in addition, but that they alone are insufficient to explain the results of this investigation.

SUMMARY

Sliding friction between metals drops from a higher static to a lower kinetic value, usually abruptly. The kinetic friction remains rather constant but shows a slow decrease as time passes, and does not instantaneously increase to its static value when the surfaces come to rest. Therefore, a simple, reversible friction-velocity relation does not exist and the friction depends rather on the history of the motion. These results support the interpretation that plasticity, adhesion, and shearing actions are involved in sliding friction of unlubricated surfaces.

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The Apparent Second-Order Transition Point of Polystyrene

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(1) At high temperatures (above 80°C) the specific volume of polystyrene is an explicit function of the temperature. $dV/dT=0.00043$. (2) At low temperatures (below 40°C) the specific volume of polystyrene depends upon its past thermal history $dV/dT=0.00024$, although V is not explicitly fixed by the temperature alone. (3) When a sample of polystyrene is cooled at constant rate, it contracts according to the higher expansion coefficient until some critical region of temperature, and then contracts according to the smaller coefficient. (4) The *position* of the critical temperature region depends upon the rate of cooling. The faster the cooling, the higher the "transition point." (5) In the intermediate temperature range (say 40°C to 80°C), complex time effects can be observed. A sample heated from 20°C to this intermediate range will first expand, according to the small expansion coefficient, and then contract slowly toward a smaller volume. The rate of this contraction is greater at higher temperatures. (6) All the observations are in fair accord with the interpretation of the "second-order transition point" of polystyrene as the temperature at which rate of volume change becomes comparable with the experimental time scale.

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MANY supercooled organic liquids possess definite short temperature ranges in which they change to "vitreous solids" or "glasses."[1,2] This transition is entirely distinct from crystallization.[1–4] There is no discontinuity in the primary thermodynamical variables, and x-ray studies show that no sudden structural change is involved. On the other hand, the first derivative variables—specific heat, coefficient of expansion, etc.—undergo marked changes in the course of a very few degrees.[5–14]

The correlation between the "transformation points" as determined by entirely different methods is very striking, and gives the impression of a true thermodynamical singularity. The discontinuities in specific heat and expansion coefficient come in the same general temperature range as that in which the mechanical "softening point" occurs.

Here, the second-order transition (glass-liquid) of polystyrene will be interpreted as being merely the point where the rate of attainment of equilibrium is of the same order of magnitude as the time-scale of ordinary physical experiments. According to this interpretation, the transformation temperature does not represent a fundamental singularity in any thermodynamical property, but is rather the point at which the internal viscosity passes through a certain arbitrary range of values.

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MECHANICAL HARDENING

The viscosity of a supercooled liquid is roughly represented by the Andrade equation:

$$\eta = Ae^{B/T}. \tag{1}$$

Although this represents a rather rapid change with temperature, there is nothing to indicate the existence of a temperature singularity.

Mechanically, the distinction between glass and liquid depends upon the time-scale of the rheological experiment used and the mechanical relaxation time of the amorphous material. The simplest relation between shearing stress, S, and deformation in shear γ for such a material is the Maxwell relation:[15]

$$\frac{d\gamma}{dt} = \frac{1}{G}\frac{dS}{dt} + \frac{1}{\eta}S \tag{2}$$

where G is the shear modulus and η the viscosity. This leads to an exponential relaxation of stress at constant deformation

$$S = S_0 e^{-t/\tau}; \quad \tau = \eta/G. \tag{3}$$

Since the relaxation-time τ is proportional to η, it must vary exponentially with $1/T$. The "hardening point" is the temperature at which τ is of the same order of magnitude as the time scale of the mechanical observation. As long as τ is much less than a second, the material will be classified as a liquid. If τ is more than several seconds, a cursory observation will result in its being termed a "glass." If a slower mechanical test is used, i.e., one with a longer time-scale, a hardening point will be observed at a somewhat lower temperature, corresponding to a lower rate of stress relaxation.

VOLUME CHANGES

Liquid structure is characterized by local "crystal-like" order, but by absence of any long range structure. The nearest neighbors of any molecule in a liquid are arranged about it in much the same manner as in the crystalline state, but molecules some eight or ten angstroms away are arranged practically at random. Such an imperfect packing requires geometrically that there

[15] J. C. Maxwell, Phil. Mag. **157**, 49 (1867).

be certain gaps or "holes" in the structure.[16–21] The greater the disorder of the molecular arrangement, the greater the number of holes.

X-ray patterns indicate that when a normal liquid cools, it takes on a more ordered, and in most cases, more compact structure. The equilibrium number of holes, and hence the free volume, decreases with decreasing temperature. However, this volume contraction cannot take place instantaneously. The attainment of the more compact structure depends upon molecular diffusion. Furthermore, no local rearrangement involving a limited number of molecules can affect the volume of the sample as a whole, but can only change the positions of the gaps or holes, and perhaps the manner in which the total free volume is distributed among the different "holes." This could be looked upon as a "diffusion of the holes," in which the identity of individual holes is not necessarily preserved, but only the total free volume. While diffusing, the "holes" can divide, or, upon meeting, combine. If the change of structure is looked upon in this way, it is clear that, in order for the process to yield a volume change, "holes" must diffuse to the surface of the sample and disappear.

At high temperatures, the diffusion process is rapid enough that volume expansions and contractions are able to attain equilibrium in a very short time. Hence the large coefficient of expansion of a liquid at high temperatures. At low temperatures, on the other hand, molecular diffusion is slow, and holes cannot diffuse to the surface in a reasonable time. The liquid is thus unable to attain its equilibrium structure, but remains "frozen" in a less compact arrangement which is stable only at some higher temperature. Such a liquid can still expand and contract, of course, by the same mechanism as a crystal lattice—that is, change in amplitude of anharmonic thermal vibrations. This mechanism of contraction requires no diffusion of matter, and hence volume-equilibrium is reached in a relatively short time. At low temperatures, the coefficient of expansion of a liquid is very close to that of the corresponding crystalline solid.

The large expansion coefficient is characteristic of the material at temperatures which are high enough for the time of relaxation into equilibrium volume to be *very short* compared to the time-scale of the density measurement. The small coefficient is characteristic at low temperatures where the time of relaxation is *very long* compared to the experimental time-scale. The transition from one coefficient to the other occurs in the temperature range where the time of relaxation is of the same order of magnitude as the experimental time-scale. The position of the transition temperature is then dependent upon the time-scale of the experimental investigation. However, in order essentially to shift the observed transition temperature, the time-scale of the experimental investigation would have to be changed to a different *order of magnitude*. Since most experimental investigations have roughly the same time-scale, the "second-order transition point" usually appears as a fairly definite, reproducible temperature—giving the appearance of a singularity.

Near the transition temperature, the time-lags in establishment of equilibrium volume are experimentally observable, and in this region rate studies as well as equilibrium studies are necessary. By interpretating the second-order transition point as the temperature where the rate of attainment of equilibrium structure fits the ordinary observational time-scale, it is possible to explain certain apparently anomalous observations in this temperature range.[16,22–28]

It is not surprising that the "transformation point," as determined by different methods, lies roughly in the same temperature range, since the time-scales for specific heat, density, and

[16] Academy of Science of Amsterdam: First and second report on Viscosity; Amsterdam 1935 and 1938.
[17] H. Eyring, J. Chem. Phys. **4**, 283 (1936).
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[24] J. Frenckel and J. Obrastzov, J. Phys. U.S.S.R. **2**, 131 (1940).
[25] A. Gemant, J. App. Phys. **12**, 680 (1941).
[26] H. R. Lillie, J. Am. Ceram. Soc. **16**, 619 (1933).
[27] R. Simha, J. App. Phys. **13**, 201 (1942).
[28] R. Simha, J. Phys. Chem. **47**, 348 (1943).

mechanical investigations will be at least comparable, and since the rate of attainment of equilibrium for all these properties depends upon the same molecular process of diffusion. Exact definition of a unique sharp temperature is impossible even upon the basis of a single property. Thus, a rapid mechanical test carried out at different temperatures will give a higher "vitrification temperature" than a slow mechanical test.

Since most ordinary mechanical tests have a somewhat shorter time-scale than most thermal measurements, one might expect reported "softening points" to be consistently higher than the transitions points determined from specific heat or density measurements.

EXPERIMENTAL

An experimental investigation was made of the volume-temperature-time relationship in polystyrene—above, below, and in the range of the second-order transition. The results agree with the situation as outlined in the previous sections. In the case of an amorphous high polymer having sufficiently high degree of polymerization, the mechanical change which occurs in the transformation range is from a glassy to a rubbery material, rather than to a simple liquid.

At about 20°C, polystyrene exhibits a small coefficient of expansion, and no appreciable time effects. At about 80°C and higher the coefficient of expansion is large, and the time effects negligible. In an intermediate range of temperature, the time effects are very important. The density of polystyrene at high temperature is simply a function of temperature; at low temperatures, it depends upon both temperature and detailed past history.

The transition, glassy to rubbery polystyrene, was investigated by means of volume changes.

A weighed sample of polystyrene with a viscosity average molecular weight around 200,000 was placed in a dilatometer of known volume. Water was used as the enclosing liquid. The dilatometer was immersed in a temperature bath of high heat capacity, which could be heated or cooled at a controlled rate, or held at any desired temperature between 20°C and 95°C (±0.2°C). Several constant temperature baths were available (±0.1°C) so that it was also possible to change the temperature of the sample quickly

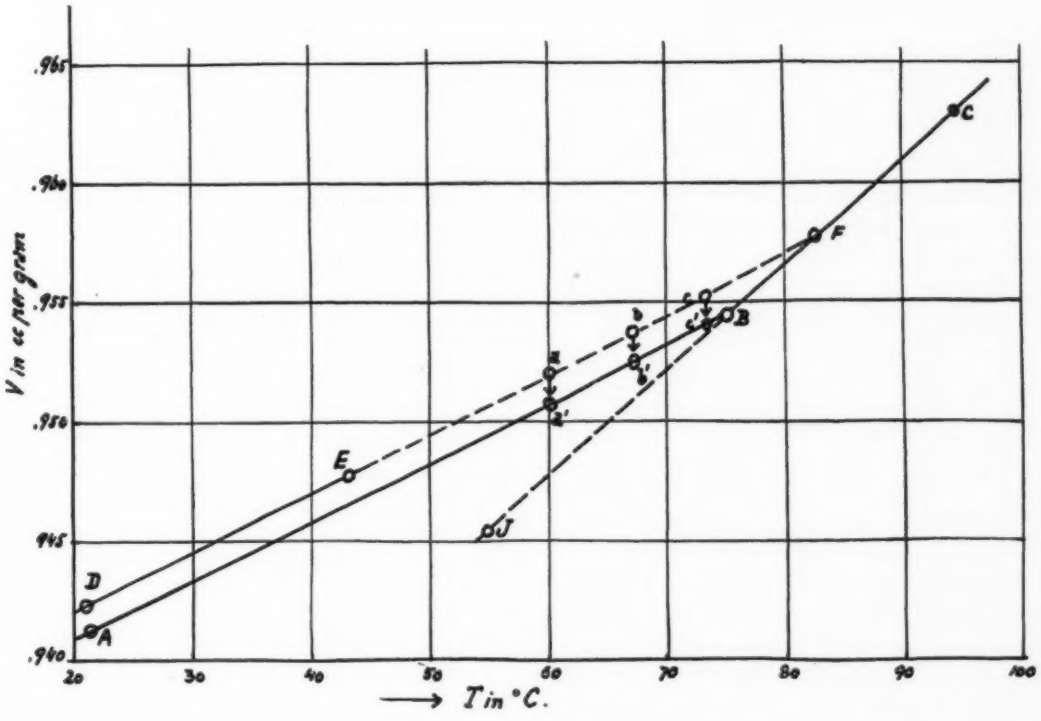

FIG. 1. V in cc per gram *vs.* temperature in °C.

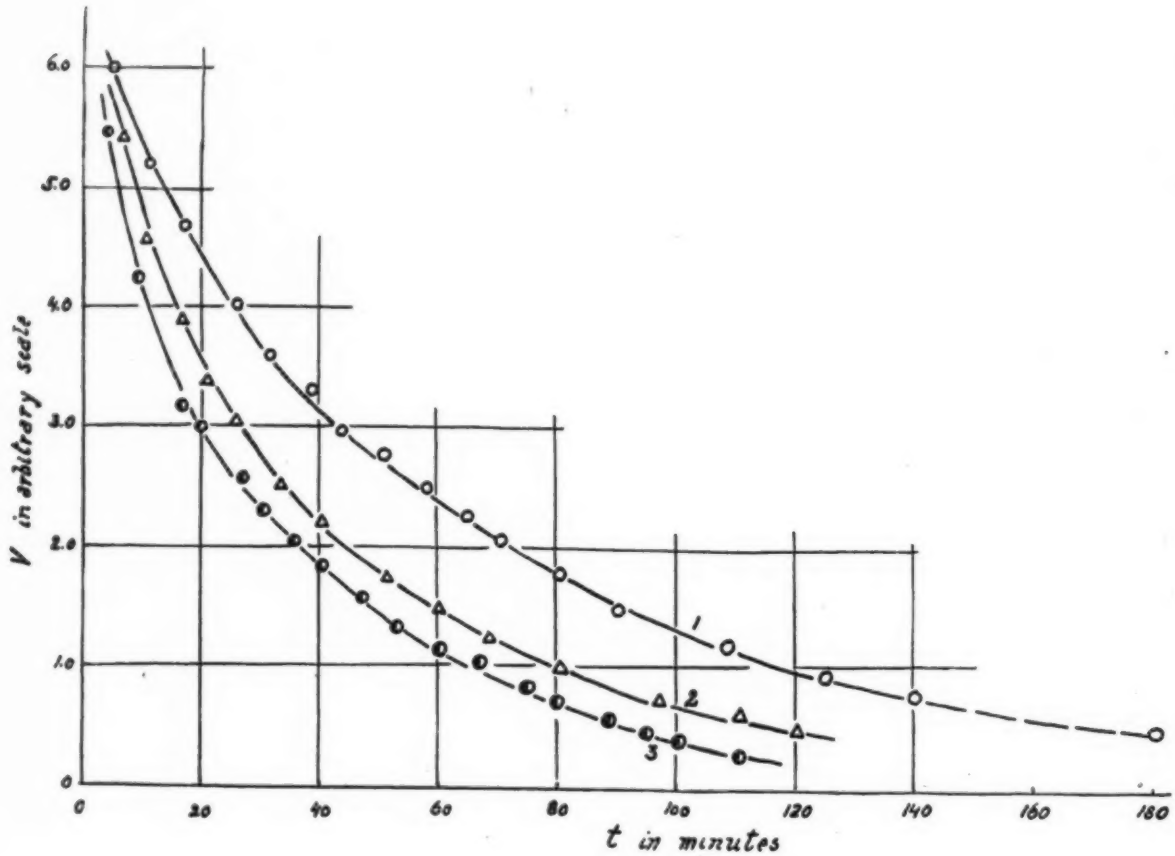

FIG. 2. V in arbitrary scale as function of time.

by a large amount by moving the dilatometer from one bath to another.

A cathetometer was used to determine the height of water in the capillary of the dilatometer. From this height the known expansion characteristics of the glass, and the known volume-temperature relation of water, it was possible to compute the specific volume of the polystyrene at any time. The precision of the volume measurements was great enough so that the limiting factor was the temperature control and measurement.

A series of temperature sequences were employed. The results are represented in Fig. 1. At high temperatures, the specific volume of the sample always followed curve BC, on heating or cooling ($dV/dT=0.00043$). When a sample was cooled from 95°C to 20°C at the rate of 0.2°C/min., the curve CBA was followed. At low temperatures (below 40°C) the curve AB was reproducible on heating and cooling ($dV/dT=.00024$). It was not possible to follow this curve back through the intermediate temperature region, however.

When a sample at 95°C was *quickly* cooled to 20°C, the volume was higher than that indicated by curve AB. The sample in this state none the less had within the limits of error the same *coefficient of expansion* ($dV/dT=0.00024$) as the sample which was slowly cooled. This is indicated by the solid line DE of Fig. 1. Upon heating and cooling, below about 40°C, the sample followed curve DE reversibly. When heated into the intermediate region, indicated by the dotted line EF, the sample deviated from the curve obtained by extrapolating DE to F. The implication is that during the rapid cooling, the sample followed the volume-temperature curve $CFED$, completely analogous to the slow cooling curve $CFBA$. This could not be directly proven, since the rate of cooling was too high.

The interpretation of these results is the following:

The equilibrium specific volume as a function of temperature is given by curve *CBJ* (the portion *BJ* being extrapolated from the experimental portion *CB*). At a high temperature (say 90°C), the sample can, by rapid segment diffusion, actually assume the equilibrium volume for that temperature in a short length of time. If a sample is cooled at an infinitesimal rate, it follows curve *CBJ*. When cooled at an appreciable rate, it follows this curve as long as the volume can take on its equilibrium value quickly enough. The *rate of volume change* decreases with decreasing temperature. At some temperature (or rather, in a narrow temperature range) the volume contraction ceases to be fast enough. At a cooling rate of 0.2°C per second, this temperature range is at about 75°C (point *B*). As the cooling is continued, a new, lower, coefficient of expansion is observed. If the sample is cooled rapidly, the deviation from the equilibrium curve comes sooner (point *F*). At any temperature, the sample tends to go from its high, non-equilibrium volume to the equilibrium volume of curve *CJ*. At very low temperatures, however, this tendency is not noticeable because of the low *rate* of the process. Thus at low temperatures the sample when heated and cooled follows the particular non-equilibrium volume curve upon which it happens to be.

A sample which had been quickly cooled at 20°C was quickly heated to 60°C and held at that temperature. The volume slowly dropped from the line *DEF* to the line *AB* along *aa'*. This volume change with time is shown in terms of an arbitrary scale on the ordinate in curve 1 of Fig. 2. It can be seen that the time required for the sample to undergo half the volume change is about 45 minutes.

When a quenched sample was heated to 67.2°C, the volume dropped along the line *bb'* in Fig. 1. The rate of this drop is indicated curve 2 in Fig. 2; the half-time is about 25 minutes.

When a quickly cooled sample was heated to 73.4°C, the volume dropped along *cc'* (Fig. 1). The rate is indicated in Fig. 2 by curve 3, having a half-time of about 20 minutes.

The volume-time relations of Fig. 2 are not simple exponential curves. It seems not possible, without a more thorough investigation, to compute from them an activation energy[19, 20, 29] for the contraction. This will be done in another paper as soon as further experiments are available.

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Erratum: The Approximate Mathematical Methods of Applied Physics as Exemplified by Application to Saint-Venant's Torsion Problem

[J. App. Phys. **14**, 469 (1943)]

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ON page 478 of the above article, in reference (1) the wrong periodical is listed. It should be the *American Journal of Physics* and not the *Journal of Applied Physics.*

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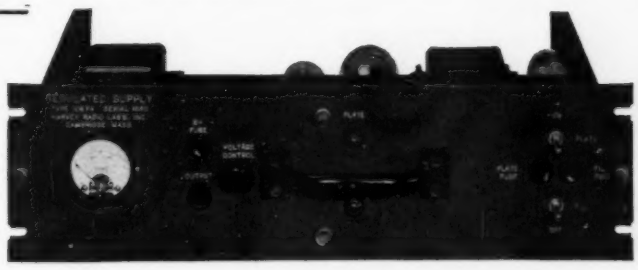

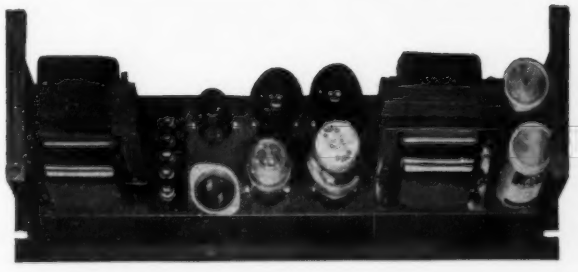

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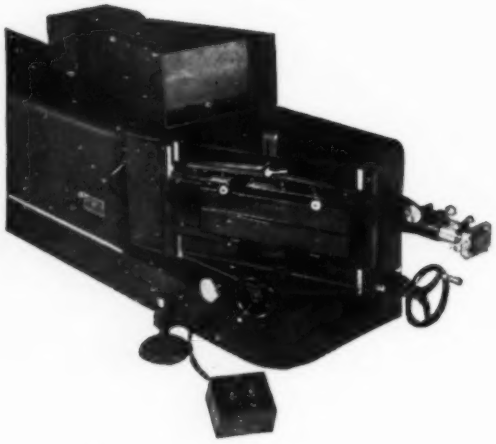

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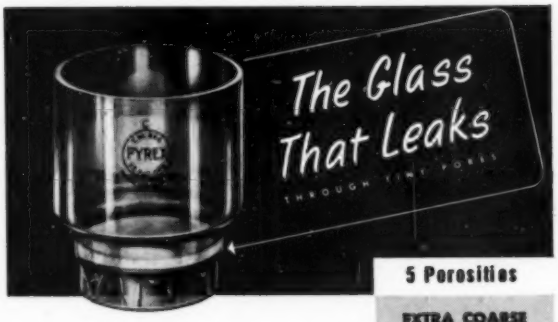

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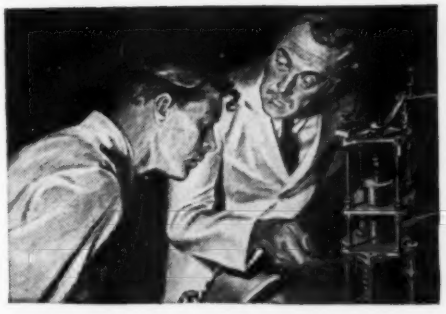

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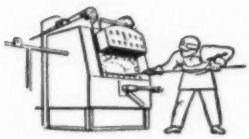

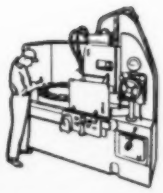

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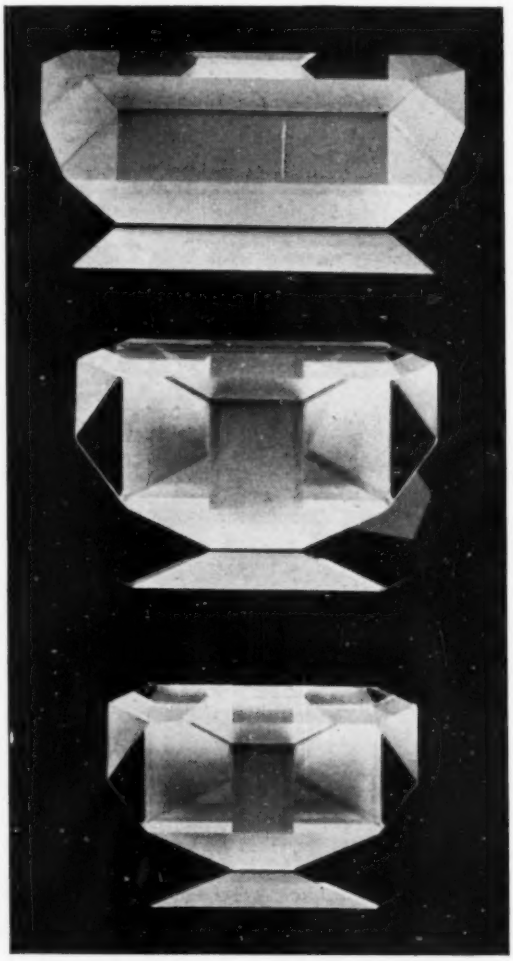

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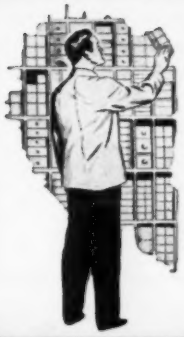

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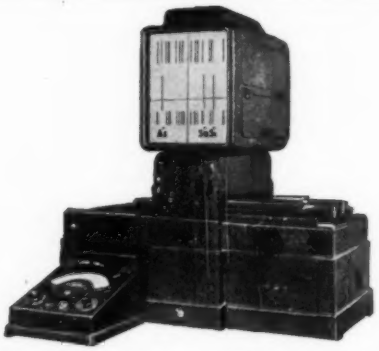

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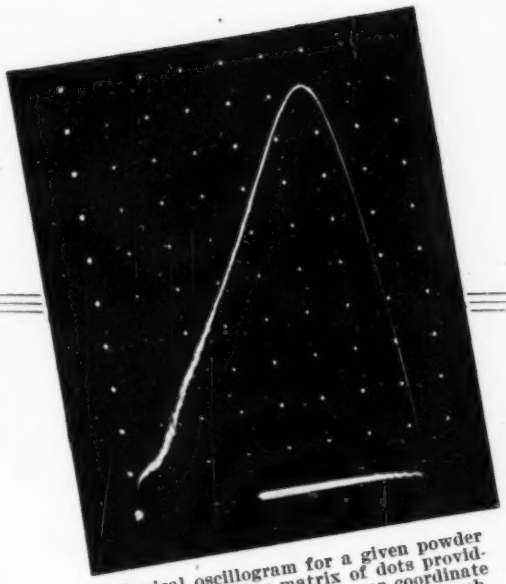

Typical oscillogram for a given powder sample, showing matrix of dots providing an accurate reference or coordinate system for the burning powder graph, permanently photographed.

Straight Shooting . . .

● The DuMont Type 235 cathode-ray oscillograph is being used in conjunction with a closed-bomb method of powder testing. Signals for the oscillograph are generated by the closed-powder bomb. Potentials furnished by burning powder provide horizontal and vertical deflection signals. Luminous dots electronically imposed on the screen provide an accurate calibration means. The resultant combination oscillogram is photographed, as shown above, for a permanent record.

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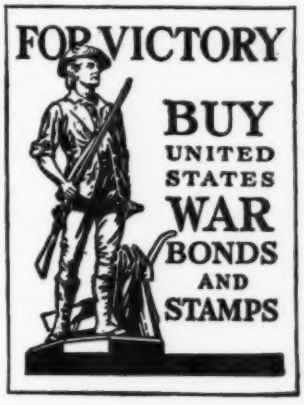

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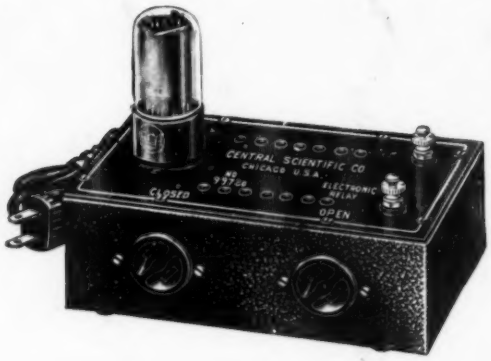

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